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January 1992

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Technical Note

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## PAINT REMOVAL USING CRYOGENIC PROCESSES

**ABSTRACT** The use of a high-pressure jet of cryogenic fluid (e.g., liquid nitrogen at  $-320^{\circ}\text{F}$ ) to remove paint and other protective coatings from Navy aircraft and ships was studied. The objective of the work was to explore the feasibility of developing a paint removal method that is less harmful to the environment than the chemical paint stripping methods presently in use. It was learned that only thick ( $t > 0.020$  inch) films of paint can be effectively removed by the mechanism of thermal shock. Aircraft paint is too thin and flexible to be removed by cryogenic methods. Cryogenic methods are not recommended for use on ships because of the danger of steel embrittlement by low temperatures. It was demonstrated that a jet of liquid nitrogen can effectively remove certain paints (regardless of thickness) by the mechanism of differential thermal contraction. The process may have application where control of paint waste is essential, for example, removal of thick films of lead base paint.

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# METRIC CONVERSION FACTORS

## Approximate Conversions to Metric Measures

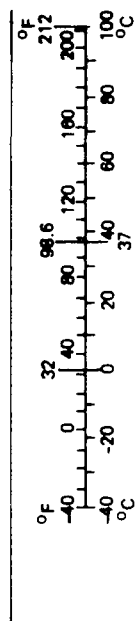
| Symbol                     | When You Know          | Multiply by                | To Find             | Symbol          |
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| <b>LENGTH</b>              |                        |                            |                     |                 |
| in                         | inches                 | 2.5                        | centimeters         | cm              |
| ft                         | feet                   | 30                         | centimeters         | cm              |
| yd                         | yards                  | 0.9                        | meters              | m               |
| mi                         | miles                  | 1.6                        | kilometers          | km              |
| <b>AREA</b>                |                        |                            |                     |                 |
| in <sup>2</sup>            | square inches          | 6.5                        | square centimeters  | cm <sup>2</sup> |
| ft <sup>2</sup>            | square feet            | 0.09                       | square meters       | m <sup>2</sup>  |
| yd <sup>2</sup>            | square yards           | 0.8                        | square meters       | m <sup>2</sup>  |
| mi <sup>2</sup>            | square miles           | 2.6                        | square kilometers   | km <sup>2</sup> |
|                            | acres                  | 0.4                        | hectares            | ha              |
| <b>MASS (weight)</b>       |                        |                            |                     |                 |
| oz                         | ounces                 | 28                         | grams               | g               |
| lb                         | pounds                 | 0.45                       | kilograms           | kg              |
|                            | short tons (2,000 lb)  | 0.9                        | tonnes              | t               |
| <b>VOLUME</b>              |                        |                            |                     |                 |
| tsp                        | teaspoons              | 5                          | milliliters         | ml              |
| Tbsp                       | tablespoons            | 15                         | milliliters         | ml              |
| fl oz                      | fluid ounces           | 30                         | milliliters         | ml              |
| c                          | cups                   | 0.24                       | liters              | l               |
| pt                         | pints                  | 0.47                       | liters              | l               |
| qt                         | quarts                 | 0.95                       | liters              | l               |
| gal                        | gallons                | 3.8                        | liters              | l               |
| ft <sup>3</sup>            | cubic feet             | 0.03                       | cubic meters        | m <sup>3</sup>  |
| yd <sup>3</sup>            | cubic yards            | 0.76                       | cubic meters        | m <sup>3</sup>  |
| <b>TEMPERATURE (exact)</b> |                        |                            |                     |                 |
| °F                         | Fahrenheit temperature | 5/9 (after subtracting 32) | Celsius temperature | °C              |

## Approximate Conversions from Metric Measures

| Symbol                     | When You Know                     | Multiply by       | To Find                | Symbol          |
|----------------------------|-----------------------------------|-------------------|------------------------|-----------------|
| <b>LENGTH</b>              |                                   |                   |                        |                 |
| mm                         | millimeters                       | 0.04              | inches                 | in              |
| cm                         | centimeters                       | 0.4               | inches                 | in              |
| m                          | meters                            | 3.3               | feet                   | ft              |
| m                          | meters                            | 1.1               | yards                  | yd              |
| km                         | kilometers                        | 0.6               | miles                  | mi              |
| <b>AREA</b>                |                                   |                   |                        |                 |
| cm <sup>2</sup>            | square centimeters                | 0.16              | square inches          | in <sup>2</sup> |
| m <sup>2</sup>             | square meters                     | 1.2               | square yards           | yd <sup>2</sup> |
| km <sup>2</sup>            | square kilometers                 | 0.4               | square miles           | mi <sup>2</sup> |
| ha                         | hectares (10,000 m <sup>2</sup> ) | 2.5               | acres                  |                 |
| <b>MASS (weight)</b>       |                                   |                   |                        |                 |
| g                          | grams                             | 0.035             | ounces                 | oz              |
| kg                         | kilograms                         | 2.2               | pounds                 | lb              |
| t                          | tonnes (1,000 kg)                 | 1.1               | short tons             |                 |
| <b>VOLUME</b>              |                                   |                   |                        |                 |
| ml                         | milliliters                       | 0.03              | fluid ounces           | fl oz           |
| l                          | liters                            | 2.1               | pints                  | pt              |
| l                          | liters                            | 1.06              | quarts                 | qt              |
| l                          | liters                            | 0.26              | gallons                | gal             |
| m <sup>3</sup>             | cubic meters                      | 35                | cubic feet             | ft <sup>3</sup> |
| m <sup>3</sup>             | cubic meters                      | 1.3               | cubic yards            | yd <sup>3</sup> |
| <b>TEMPERATURE (exact)</b> |                                   |                   |                        |                 |
| °C                         | Celsius temperature               | 9/5 (then add 32) | Fahrenheit temperature | °F              |



\*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price \$2.25, SD Catalog No. C13.10 286.



| REPORT DOCUMENTATION PAGE  |   |  | Form Approved<br>OMB No. 0704-018                            |  |
|--|---|--|--|--|
| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.   |   |  |  |  |
| 1. AGENCY USE ONLY (Leave blank)   |   | 2. REPORT DATE<br><br>January 1992                             |  | 3. REPORT TYPE AND DATES COVERED<br><br>Final: Oct 1989 through Jun 1991 |
| 4. TITLE AND SUBTITLE<br><br>PAINT REMOVAL USING CRYOGENIC PROCESSES   |   |  | 5. FUNDING NUMBERS<br><br>PR - RM33E80-D4<br>WU - DN669045   |  |
| 6. AUTHOR(S)<br><br>Richard E. Kirts and Philip L. Stone   |   |  |  |  |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)<br><br>Naval Civil Engineering Laboratory<br>Port Hueneme, CA 93043-5003  |   |  | 8. PERFORMING ORGANIZATION<br>REPORT NUMBER<br><br>TN - 1839 |  |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)<br><br>Office of the Chief of Naval Research<br>Office of Naval Technology<br>Arlington, VA 22217-5000   |   |  | 10. SPONSORING/MONITORING<br>AGENCY REPORT NUMBER            |  |
| 11. SUPPLEMENTARY NOTES  |   |  |  |  |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT<br><br>Approved for public release; distribution unlimited.   |   |  | 12b. DISTRIBUTION CODE                                       |  |
| 13. ABSTRACT (Maximum 200 words)<br><br>The use of a high-pressure jet of cryogenic fluid (e.g., liquid nitrogen at -320°F) to remove paint and other protective coatings from Navy aircraft and ships was studied. The objective of the work was to explore the feasibility of developing a paint removal method that is less harmful to the environment than the chemical paint stripping methods presently in use. It was learned that only thick ( $t > 0.020$ inch) films of paint can be effectively removed by the mechanism of thermal shock. Aircraft paint is too thin and flexible to be removed by cryogenic methods. Cryogenic methods are not recommended for use on ships because of the danger of steel embrittlement by low temperatures. It was demonstrated that a jet of liquid nitrogen can effectively remove certain paints (regardless of thickness) by the mechanism of differential thermal contraction. The process may have application where control of paint waste is essential, for example, removal of thick films of lead base paint. |   |  |  |  |
| 14. SUBJECT TERMS<br><br>Paint removal, paint stripping, depainting  |   |  | 15. NUMBER OF PAGES<br><br>48                                |  |
|  |   |  | 16. PRICE CODE   |  |
| 17. SECURITY CLASSIFICATION<br>OF REPORT<br><br>Unclassified   | 18. SECURITY CLASSIFICATION<br>OF THIS PAGE<br><br>Unclassified | 19. SECURITY CLASSIFICATION<br>OF ABSTRACT<br><br>Unclassified | 20. LIMITATION OF ABSTRACT<br><br>UL                         |  |

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## INTRODUCTION

This report discusses the feasibility of using liquid nitrogen to remove paint and other protective coatings from Navy aircraft and ships. The objective of this study was to explore the feasibility of developing a paint removal method that is less harmful to the environment than the chemical paint stripping methods presently in use.

## BACKGROUND

Paint and other protective coatings must be periodically removed, or stripped, from aircraft and ship hulls so that: (1) the hulls can be inspected for corrosion and other forms of damage, or (2) to facilitate repairs or modifications. At the present time, many of the methods used to remove paint from aircraft and ships generate large quantities of hazardous waste. Aircraft are often stripped of paint using chemicals that soften the paint film so that it can be scraped or rinsed off. These chemical strippers are usually a combination of methylene chloride, organic acids, phenols, and waxes. The used stripper, paint sludge, and water used to rinse the aircraft must be treated as hazardous waste material.

It is estimated that the Navy spends in excess of \$30 M per year to dispose of 10 million gallons of liquid waste from aircraft paint stripping operations. Ship hulls, being made of a thicker material, are usually stripped of paint using abrasive blasting or chipping methods. Heavy metals in the spent blasting grit and the dust and noise generated by the blasting process have been reported problems at several activities. Therefore, a need exists for a new method of stripping paint from Navy hulls that minimizes the impact on the environment.

This research was suggested by the successful use of a jet of liquid nitrogen to remove old paint and other coatings from the inside surfaces of the Statue of Liberty during its restoration in the early 1980s (Refs 1, 2, and 3). The National Park Service needed to develop a method of paint removal that would accomplish several goals. First, the paint removal method had to be environmentally benign. Second, the method had to be safe and easy to use, especially in an enclosed space. Finally, the method could not harm the Statue in any manner. The use of a high-pressure jet of liquid nitrogen to remove paint was suggested by an engineer from the National Park Service, and developed with labor and materials donated by the Linde Division of the Union Carbide Corporation. The system developed by the Park Service was operated in the same manner as a high-pressure water jet cleaning system. In this case, the working fluid, liquid nitrogen, quickly boils away after being sprayed onto the surface - leaving only chips of paint to be cleaned up.

The National Park Service performed extensive metallurgical tests on copper alloys subjected to the cryogenic paint stripping process and determined that the process would do no harm to the Statue.

The evidence that the cryogenic paint stripping process is environmentally safe, is simple and easy to use, and that the Statue's thin copper skin is thermally and structurally similar to an aluminum aircraft skin suggested that cryogenic paint stripping methods might be applied with equal effectiveness to Navy aircraft. The additional facts that the Park Service reported high paint removal efficiency (about 3 square feet per gallon of liquid nitrogen), that liquid nitrogen is relatively inexpensive (about \$2 per gallon in small quantities), and that liquid nitrogen is a substance the Navy already uses for several industrial processes, also suggest that a practical, cost-effective cryogenic paint removal process could be developed for Navy use.

## DISCUSSION

### Objective

The objective of this study was to determine the feasibility of using liquid nitrogen, alone or in combination with other substances, to remove paint and other protective coatings from Navy aircraft and ship hulls.

### Approach

This study was primarily experimental in nature, but was guided by limited theoretical work on the mechanisms of paint removal by cryogenic processes.

### Theory

There are several possible ways that the application of liquid nitrogen or other cryogenic substances might remove paint and other surface coatings. Three possible paint removal mechanisms are explained in the following paragraphs.

**Thermal Shock.** If the surface of a material is suddenly made much colder than the interior of the material, the temperature difference results in a tensile stress on the surface equal to (Ref 4):

$$\sigma = \alpha \cdot E \cdot \Delta T / (1 - \nu) \quad (1)$$

where:  $\sigma$  = surface stress, psi

$\alpha$  = coefficient of thermal expansion,  $1/^{\circ}\text{F}$

$E$  = modulus of elasticity, psi

$\Delta T$  = difference in temperature between surface and substrate,  $^{\circ}\text{F}$

$\nu$  = Poisson's ratio

If this stress is greater than the tensile strength of the material, the material will develop a network of surface cracks. Because the surface is in tension, the material between the cracks should curl upward at the edges. The cracked surface and curled edges of the coating weaken the attachment of the coating to the substrate, so that the removal can be more easily accomplished by the force of the fluid jet or by some mechanical device, such as a stiff bristle brush.

Substituting numerical values for the variables in Equation 1 (using handbook data for acrylic plastic, Reference 5) gives:

$$\sigma = \frac{50 \text{ E-06} \cdot 400,000 \cdot 400}{(1.0 - 0.4)} = 13,000 \text{ psi}$$

Reference 6 reports ultimate tensile strength values of 10,500 to 13,000 psi for many plastic materials. Therefore, damage to a painted surface by the mechanism of thermal shock seems possible.

S.S. Manson (Ref 7) assumed that the stress at any point in a material can be described by a linear relationship with temperature:

$$\sigma' = (T_{\text{avg}} - T)/T_0 \quad (2)$$

where  $\sigma'$  is the ratio of the stress developed in a thermally shocked material to the stress that would be developed with no thermal shock. Thus,

$$\sigma' = \sigma \cdot (1 - \nu)/(E \cdot \alpha \cdot T_0) \quad (3)$$

Surface stresses were obtained by Manson from Equations 2 and 3 by determining the average temperature and how the surface temperature varies with time. Manson's entire solution for the surface stress in a flat plate is presented in Figure 1. In Figure 1,

$\sigma'$  = nondimensional stress as defined in Equation 3

$\beta$  = nondimensional heat transfer rate =  $a \cdot h/k$

$\theta$  = nondimensional time =  $k \cdot t/\rho \cdot c_p \cdot a^2$

In the above equations,

$a$  = one half the plate thickness, ft

$h$  = heat transfer coefficient, Btu/sec/sq ft/°F

$k$  = thermal conductivity, Btu/sec/ft/°F

$t$  = time, sec

$\rho$  = density, lb/cu ft

$c_p$  = specific heat capacity, Btu/lb/°F

Note in Figure 1 that to achieve a high value of thermally-induced surface stress, the heat transfer rate must be high and the duration of the heat transfer process must be brief.

Resistance to damage by thermal shock is measured by a thermal shock parameter. There are several thermal shock parameter models; one of the most accepted is that of Schott and Winkelman (Ref 8), that is:

$$P = \frac{\sigma}{E \cdot \alpha} \left[ \frac{k}{\rho \cdot c_p} \right]^{\frac{1}{2}} \quad (4)$$

where:  $P$  = thermal shock parameter

$\sigma$  = ultimate stress

$E$  = modulus of elasticity

$\alpha$  = coefficient of expansion

$k$  = thermal conductivity

$\rho$  = density

$c_p$  = heat capacity

Substituting typical values of material properties for plastic materials and aluminum alloys into Equation 4 gives a value of  $P$  for plastic materials of about 25 and a value of  $P$  for aluminum alloys of about 700. This indicates that the paint coatings should be damaged by thermal shock before the aluminum substrate is damaged.

**Differential Thermal Contraction.** The second possible mechanism of coating removal is failure of the bond between coating and substrate due to a difference in tensile stresses at the coating-substrate interface. If both coating and substrate are cooled to the same temperature, but are constrained from movement, stresses are developed within the two materials. These stresses are a function of the properties of the two materials and the temperature to which they are cooled. It can be shown that the stress developed in the coating layer of a coating-substrate system when the system is cooled is:

$$\sigma_c = \frac{(\alpha_s - \alpha_c) \cdot \Delta T \cdot E_c}{\left( 1 + \frac{t_c \cdot E_c}{t_s \cdot E_s} \right)} \quad (5)$$



where:  $\alpha$  = coefficient of thermal expansion, in./in./°F

$\Delta T$  = temperature difference, °F

$t$  = thickness, in.

$E$  = modulus of elasticity, lb/in.<sup>2</sup>

Subscripts:  $c$  = coating

$s$  = substrate

Substituting typical values for material properties and a value of temperature difference of -400°F yields a value of 18,000-psi tensile stress in the coating and 850-psi compressive stress in the aluminum substrate. Since the shear strength of acrylics is about 16,000 psi, the paint should fail at the paint-substrate interface.

Paint removal by differential thermal contraction and thermal shock may be aided by the fact that most organic materials become brittle at low temperature.

**Mechanical Forces.** Removal of a weakened or damaged paint film is aided by the forces applied to the painted surface by the jet of liquid nitrogen. If the fluid jet is steady, the force ( $F$ ) imparted on the surface will be proportional to the product of the fluid density and the square of the velocity of the jet:

$$F \propto \rho \cdot V^2 \cdot \cos(\psi) \quad (6)$$

where  $\psi$  = angle between jet and surface.

If the fluid jet consists of a time series of drops of liquid nitrogen (rather than a continuous stream) the force on the surface will be proportional to the product of the fluid density, velocity of the jet, and the speed of sound ( $C$ ) in liquid nitrogen:

$$F \propto \rho \cdot V \cdot C \quad (7)$$

The speed of sound in liquid nitrogen (about 2,700 ft/sec) is substantially greater than the speed of the fluid jet (500 ft/sec or less). Therefore, high-impact forces can be developed on the surface by pulsing the flow of liquid nitrogen rather than using a continuous stream. Pulsed flow may also offer the advantage of higher heat transfer rate from the surface than is achievable with steady flow.

**Summary of Paint Removal Mechanisms.** Review of the above three possible paint removal mechanisms suggests that the thermal shock mechanism of coating removal would use the least amount of liquid nitrogen and cool the substrate material the least. A potential problem in utilizing the mechanism of thermal shock is the difficulty of developing a high temperature gradient across a very thin layer of paint. Figure 1

shows that to develop a specific level of stress in thin paint films requires proportionally greater rates of heat transfer (e.g., if the thickness of the paint film is halved, the heat transfer rate must double). Heat transfer rates sufficiently high to develop high stress in the thin paint films found on aircraft might be difficult to achieve.

Paint film removal by the mechanism of differential thermal contraction should work on paint films of any thickness, but the method requires cooling both the paint film and the surface of the substrate material to the same low temperature. Therefore, differential thermal contraction can be expected to consume more liquid nitrogen than the mechanism of thermal shock.

Removal of paint by purely mechanical action of the fluid jet is the least attractive of the alternative paint removal mechanisms because it would consume the most liquid nitrogen, cool the substrate material the most, and possibly cause damage to thin metal and composite substrates.

## **Experimental Investigation**

**Thermal Shock.** The experimental investigation was performed in several phases. Each phase of the work developed out of the findings of the previous efforts.

**Phase I - Low Pressure, Hand-Held Applicator.** The initial experiments were performed using a simple hand-held liquid nitrogen applicator (Figure 2). The liquid nitrogen supply pressure was varied by changing the setting of the pressure relief valve on the liquid nitrogen storage flask. Liquid nitrogen was applied at flask pressures of 50, 100, 150, and 250 psig. The design of the liquid nitrogen storage flask limited flask pressure to a maximum of 250 psig. Instrumentation consisted of painted panels with pairs of strain gages and thermocouples attached to the substrate material and to the paint surface. Output from the gages was connected to a high-speed strip chart recorder. Test samples included painted aluminum, steel, fiberglass-reinforced plastic, wood, and masonry. Several different sizes and designs of nozzles were also tested.

The results of these initial tests were inconclusive. Some painted samples were not affected by the spray of cryogenic fluid, while the coating on other samples was substantially cracked, chipped, or removed. The effect of the cryogenic spray on the painted surface (in the form of cracked, chipped, or removed paint) appeared to increase as the applicator pressure was increased. Paint removal effectiveness also appeared to be very dependent on paint formulation.

The results of instrumented tests were difficult to interpret because it was very difficult to correlate the recorded strains and temperatures with the activity of the applicator operator (e.g., the speed, angle, and direction at which the applicator wand was moved).

A photograph of one of the Phase I test samples is presented as Figure 3, which shows numerous tensile stress cracks and some paint chip removal.

**Phase II - Low Pressure, Robotic Applicator.** The results of the Phase I tests showed that if cryogenic paint stripping was going to work on standard aircraft and ship hull paint systems, a more complete understanding of the mechanics of the process was required. Specifically, it was necessary to determine what application conditions, such as jet angle to the surface, distance from the nozzle to the surface, and jet translation velocity, are required to develop high strain in the paint film. As it is very difficult to control some of these variables with manual application, an automated applicator system was assembled. The automated applicator system is shown in Figure 4. The system consists of a six-axis industrial robot fitted with an adjustable liquid nitrogen applicator, an adjustable test stand with powered exhaust, a high-speed data logger, and a high-capacity data analysis computer and associated software. Use of a robotic applicator permits accurate, repeatable, rapid, and safe performance of liquid nitrogen paint stripping experiments. The robotic applicator system was assembled from surplus equipment from previous projects at the Naval Civil Engineering Laboratory (NCEL).

The robot was used to facilitate the experimental investigation of process variables by programming it to follow a path over an instrumented painted test panel. Electrical signals generated by the robot controller at appropriate points in the tool path started and stopped reading of the strain and temperature gages by the data acquisition system. Data on strain and temperature stored in the memory of the data acquisition system were then downloaded to the data analysis computer by means of telecommunications software and a null modem. Next, the raw strain data were processed by software developed at NCEL that: (1) made corrections for temperature and coefficient of thermal expansion effects (a discussion of the required corrections to strain gage data is presented in the Appendix), (2) found maximum and minimum values of strain and temperature, (3) calculated rates of change of strain and temperature, and (4) displayed the results on a color video monitor. Data from each experiment were saved as a separate disk file as a permanent record of the experiment.

Typical strain and temperature histories from experiments performed under optimized applicator conditions are presented in Figures 5, 6, and 7 for epoxy paint on aluminum, composite, and steel substrates.

Several process variables that could have an influence on the magnitude of the peak strain developed in a paint film by the application of liquid nitrogen were identified and systematically evaluated. These variables were: (1) distance from the tip of the applicator to the surface (or standoff distance), (2) angle of the jet relative to the surface (impingement angle), (3) direction of movement of the applicator relative to the direction of the jet (i.e., whether the direction of the jet is the same as the direction of movement of the applicator), (4) fluid pressure at the applicator, (5) speed of application, (6) nozzle type and diameter, and (7) initial temperature of the test panel.

Nonprocess variables include paint type and thickness, and substrate material and thickness.

The experimental method used to evaluate the relative importance of each of the variables listed above was to design a baseline set of applicator variables (speed, standoff distance, supply pressure, etc.), then systematically vary one parameter at a time - keeping the other parameters

equal to their baseline values. This method yields curves of the change in peak strain and maximum strain rate as a function of the specific variable - other variables being held constant.

The apparatus and method described above was used to perform more than 270 experiments on painted aluminum, composite, and steel test panels using the low-pressure (250-psig maximum pressure) liquid nitrogen supplied directly from a storage flask.

Typical results from these process sensitivity studies are presented in Figures 8, 9, 10, and 11.

The force produced on the aircraft surface by several different nozzle types was also measured using specially designed and constructed instrumentation. The results are presented in Figure 12 for a nitrogen supply pressure of 100 psi.

It was determined that a high speed of application, low angle of the fluid jet relative to the surface, high applicator pressure, and high initial panel temperature result in maximum peak strain in the paint layer. Peak strain rates and cooling rates were also usually found under these conditions. Strain rates as high as 30,000  $\mu\text{in./in./sec}$  and cooling rates as high as 2,000°F/sec were observed.

Based on these results, the robot was reprogrammed to operate the liquid nitrogen applicator at the optimum combination of applicator speed, angle, standoff distance, etc., and another series of experiments was performed on uninstrumented panels. The results of this series of experiments showed that thermal shock due to application of liquid nitrogen could severely crack and partially remove relatively thick (15 to 17 mil) epoxy paint applied over a primed aluminum panel.

Figure 13 is a plot of nondimensional strain versus nondimensional time, with cooling rate (which is proportional to nondimensional heat transfer rate) as a parameter. The experimental results presented in Figure 13 can be compared to the theoretical model presented in Figure 1. Experimental confirmation of the model for the thermal shock paint stripping process implies that achievement of very high heat transfer rates will be required if the thermal shock mechanism is to remove the very thin (2 to 4 mil) paint systems found on modern aircraft.

Based on the theoretical model for thermal shock and confirming experimental work, the rate of heat removal from the painted surface might have to be increased by a factor of 8 to 10 times if the mechanism of paint removal by thermal shock is to work on paint films 2 to 4 mils thick.

The most practical method of increasing the heat transfer rate between and moving fluid and a solid surface is to increase the velocity of the fluid. This is done by increasing the applicator pressure, since the speed of the jet issuing from a nozzle is proportional to the square root of the pressure drop across the nozzle.

**Phase III - Intermediate Pressure, Robotic Applicator.** A new applicator system was designed and constructed to supply liquid nitrogen to the nozzle at pressures up to 2,500 psig. The intermediate pressure applicator is shown schematically in Figure 14 and pictured in Figure 15. The applicator consists of an insulated cylinder with ellipsoidal end caps welded to each end. The cylinder, end caps, and other fittings are made of type 304 stainless steel. The applicator is filled with

liquid nitrogen at low pressure through valve A. When the cylinder is full (it holds about 1 cubic foot or 50 pounds of liquid nitrogen), valve A is closed and the cylinder is pressurized with helium gas from a high-pressure (6,000-psig) gas cylinder through pressure regulator B and valve C. The pressure regulator can be set to supply liquid nitrogen at any pressure up to 2,500 psi. Liquid nitrogen is discharged from the applicator through electrically actuated valve D. The entire intermediate pressure applicator system sits on an electronic load cell transducer that measures and displays the weight of liquid nitrogen in the cylinder. A pressure transducer fitted to the nozzle apparatus measures and displays the pressure of the liquid nitrogen at the nozzle. Both weight and nozzle pressure data are also automatically recorded by the data acquisition system.

Intermediate pressure experiments were done at 500, 1,000, 1,500, and 2,500 psi in the liquid nitrogen flask. At 1,000 psi applicator pressure, paint 8 mils in thickness was removed, but no further improvement was observed at higher application pressures. Paint films 2 to 4 mils thick were not visibly affected by the liquid nitrogen jet.

Typical experimental results from a high-pressure test are presented in Figure 16.

### Limiting Factors

Experimental results show that the magnitude of the strain developed by thermal shock to the paint film is limited in practice by two factors: (1) the magnitude of the temperature differential that can be developed across thin paint films, and (2) the rate at which heat can be transferred from the paint surface.

Figure 17 illustrates the first limiting factor: maintenance of a high-temperature gradient across the paint film. The ideal paint for the cryogenic paint stripping would be an ideal insulator, so that the surface of the paint could be cooled to  $-320^{\circ}\text{F}$  while the interior of the film remained at ambient temperature. Figure 17, however, shows that instead of the anticipated temperature difference across a paint film of about  $400^{\circ}\text{F}$ , the actual temperature difference is only about  $175^{\circ}\text{F}$ . Thus, the stress developed in the paint film will be less than half the value originally predicted. This is due to two factors: (1) paint is not an especially good thermal insulator, and (2) since the rate of heat conduction through the paint film is inversely proportional to the thickness of the film, the thinner the paint film, the more difficult it is to develop a large temperature difference across the film.

Figure 18 illustrates the second factor: maintaining a high rate of heat transfer from the test panel. When a jet of liquid nitrogen first contacts the (relatively) hot test surface of the test panel, heat is conducted out of the panel and into liquid nitrogen for a very brief time (region A in Figure 18). Very quickly, however, the liquid nitrogen starts to boil vigorously, with at first a few, then many, nucleation sites on the surface. The heat transfer rate associated with the convection of bubbles of vapor from the surface of the plate is very high (compared to conduction through liquid nitrogen) and the surface temperature drops very rapidly (region B). Soon, however, boiling occurs at so many nucleation sites that the surface is effectively covered by a film of nitrogen gas. The heat transfer rate, and therefore the cooling rate,

drop dramatically in this regime of film boiling (region C in Figure 18). The heat transfer rate is low in film boiling because the layer of nitrogen gas at the surface is a relatively poor conductor of heat. The rate of heat removal from the surface in the film boiling regime is about the same as the rate of heat addition to the surface by conduction of heat from the surrounding substrate material, so the temperature of paint film decreases much more slowly, remains about the same, or may even start to slowly increase. When the heat transfer mechanism changes from nucleate boiling to film boiling, the thermally-induced strain reaches its maximum value. As the surface cools more slowly in the region of film boiling, the magnitude of the thermal strain decreases. The thermally-induced strain is reduced to zero as the temperature difference across the paint film disappears. Clearly, a sustained rise in thermally-induced strain requires a sustained and rapid decrease in surface temperature and the central issue is how to suppress or delay the transition to film boiling.

It was initially proposed that increasing the jet velocity (by increasing the nitrogen supply pressure) would result in a sufficient increase in heat transfer rate to permit the surface temperature to drop to its approximate minimum value (-320°F) before the onset of film boiling. Experiments proved that this does not happen (at least at supply pressures of 2,500 psi or less).

### Differential Thermal Contraction

Paint removal by the mechanism of differential thermal contraction did not work on aircraft paint, although it works quite well on other paint formulations. The reason that paint removal by differential thermal contraction does not work on aircraft paint is that the effective coefficient of thermal expansion of aircraft paint is quite close to that of aluminum. Thus, the stress produced in the paint layer:

$$\sigma_c \approx (\alpha_s - \alpha_c) \cdot \Delta T \cdot E_c \quad (8)$$

is quite small. The effective coefficients of thermal expansion for several current aircraft paint formulations were carefully measured. The results are presented in Table 1. The method used to measure coefficient of thermal expansion is described in the Appendix.

One common paint formulation that is easily removed by the mechanism of differential thermal contraction is red oxide (iron oxide) primer. Even a thin layer of red oxide primer applied between the anti-corrosion coat and the topcoat resulted in effective removal of the topcoat. It was apparent that the paint removal mechanism was differential thermal contraction rather than thermal shock because: (1) the failure of the paint film was not nearly instantaneous (as it is with thermal shock) but required several seconds of liquid nitrogen application to cool the paint and the substrate both to a low temperature, (2) the process works at low pressures and applicator speed, and (3) examination of the paint chips indicated failure of the paint system was by shear at the topcoat - red oxide boundary. Figure 19 is a photograph of a panel

stripped of paint by the mechanism of differential thermal contraction. Note that, although most of the paint has been removed, there remains a residue of paint particles that must be removed by some other method.

### Solid Carbon Dioxide Abrasive Blasting

Carbon dioxide in its solid form, "dry ice," has been successfully used to strip paint from aircraft hulls and other structures. Solid carbon dioxide paint stripping systems are abrasive bead blasting systems that use small particles of dry ice as the abrasive grit. The main advantage of solid carbon dioxide paint stripping systems is the same as that of a liquid nitrogen paint stripping system: the stripping medium quickly vaporizes, leaving only paint chips to clean up. Conventional carbon dioxide paint stripping systems work by forming solid  $\text{CO}_2$  into small hard pellets, accelerating the pellets to a high velocity using compressed air, then directing the pellets against the surface to be cleaned. The pellets of solid  $\text{CO}_2$  are formed by expanding high-pressure liquid  $\text{CO}_2$  to atmospheric pressure, thereby forming a mixture of about 40 percent solid  $\text{CO}_2$  "snow" and 60 percent  $\text{CO}_2$  gas. The  $\text{CO}_2$  snow is forced by a ram through a series of dies that determine the size of the solid  $\text{CO}_2$  pellets and, to some extent, the hardness of the pellets.

An apparatus was designed, built, and tested which combines the thermal shock effect of the liquid nitrogen paint stripping concept with the mechanical abrasion effect of solid carbon dioxide particles. The system process and hardware are illustrated in Figures 20 and 21. High-pressure liquid carbon dioxide (point A in Figures 20 and 21) is first cooled by cold nitrogen gas to a temperature of approximately  $-50^\circ\text{F}$  (point B). Next, the cold liquid carbon dioxide is slowly reduced in pressure to about 150 psi (point C). The cold liquid  $\text{CO}_2$  is then injected into a chamber filled with cold nitrogen gas at 150 psf. When the liquid  $\text{CO}_2$  enters the chamber, one of two things will occur: the liquid filament will break up into droplets which will freeze into solid particles while forming some  $\text{CO}_2$  gas, or the liquid filament will be frozen and broken into small particles by the surrounding atmosphere of cold nitrogen gas. The surrounding cold nitrogen gas may also increase the hardness of the  $\text{CO}_2$  particles. The mixture of cold nitrogen and carbon dioxide gases and particles of solid carbon dioxide are accelerated to a very high velocity through the convergent-divergent nozzle at the end of the mixing chamber. This cold, high-velocity stream of particles of solid carbon dioxide is directed at the surface to be cleaned. The size of the solid particles is controlled by the diameter of the hole in the injector tube, the number of solid particles is controlled by the number of holes in the injector tubes, and the velocity of the particles is controlled by the area ratio of the convergent-divergent nozzle cross sections.

The device described above was designed, fabricated, and tested. Numerous modifications to the basic design were also evaluated. The system produced a high-velocity stream of small particles of solid carbon dioxide, but was not effective at removing paint from sample aircraft panels. The small particles of solid carbon dioxide were too soft to abrade hard aircraft coatings. Also, the particles of solid carbon dioxide tended to stick to the painted surface, forming a coating of solid carbon dioxide on the surface that protected the paint from further particle impact.

## Materials Studies

**Paint.** An understanding of the mechanisms and limitations of the cryogenic paint stripping process requires knowledge of the basic mechanical properties of paint at very low temperature. Approximately 40 tests were performed to determine the tensile properties of aircraft paint at cryogenic temperatures. The tests were performed using a computer controlled tensile testing machine (an Instron model 1122) equipped with a strip chart recorder to plot sample extension versus force. Paint samples were prepared by painting a sheet of plastic that had been coated with a silicone release agent. After the paint dried, the film of paint was peeled from the plastic backing then cut into uniform 20-mm wide strips with a cutting device designed for preparing paint samples. An Instron environmental chamber was fitted to the tensile testing machine to keep the paint samples at a uniform cold temperature. The environmental chamber is an insulated cabinet which fits between the base and the drawbar of the tensile testing machine. The temperature controller regulates the flow of liquid nitrogen to keep the temperature in the chamber at a preset value.

The paint tensile tests were conducted at -250°F. Tensile tests were also performed at room temperature. Extension rates of 5, 10, and 20 mm/min were used in the tests to determine if the tensile properties were dependent on strain rate. A typical result of the tensile test experiments is presented in Figure 22. Most of the stress-strain curves are characterized by two distinct types of material behavior. As the load is first applied to the paint, the cold paint film stretches elastically, with a very low value of modulus of elasticity (E). Then, following a strain of about 2 percent, the behavior of the paint film changes to that of a material having a much higher value of E. Figure 23 presents the observed strain at rupture versus the speed of extension for some of the tensile tests performed at cryogenic temperature. Paint samples at room temperature typically stretched 35 to 45 percent before breaking.

The gage length of the strain gages used in the measurement of thermal shock-induced strain was 0.125 inch. Therefore, a typical measured peak value of thermally-induced strain of 3,000  $\mu\text{in./in.}$  can be expressed as a percentage of gage length as:

$$\% \text{ strain (peak)} = \frac{2500 \text{ E-06} \cdot 100 \%}{0.125} = 2\% \quad (9)$$

Peak recorded strain was about 2.4 percent. This indicates that for most of the thermal shock experiments the paint film was not strained enough to transition to high E behavior (i.e., developed strains were to the left of the knee in the data in Figure 20, or about 2 percent strain).

**Metals.** The primary concern regarding the use of the cryogenic paint stripping method on painted metal surfaces is the transition from a ductile state to a brittle state that occurs in some metals at low temperature. Although a brittle material may be strong, the fact that it is brittle implies that any material failure will be sudden and



catastrophic rather than a gradual yielding (which occurs in ductile materials). The loss of ductility at low temperatures is usually measured by a test that determines the impact energy required to produce material failure by brittle fracture. Commonly used impact energy tests are the Charpy V and keyhole notch tests, the Izod test, and the Navy Research Laboratory drop test.

A review of references on cryogenic metallurgy indicates that metals having a face-centered cubic crystal structure such as copper, aluminum, and nickel do not undergo a marked decrease in ductility as the temperature is decreased.

The low-temperature elastic properties of ferrous metals are dependent on the deoxidation process used in manufacture, heat treatment, and alloy content. Steels deoxidized using aluminum have a fine grain structure which is more resistant to brittle fracture. Quenched and tempered steels have better low-temperature impact resistance than annealed or soft steels. Alloying steel with nickel, chromium, and manganese provides the greatest gain in impact resistance at low temperature. Low-carbon 3 percent nickel steel has impact strength to  $-100^{\circ}\text{F}$ , low-carbon 9 percent nickel steel and maraging (high nickel) steel to  $-320^{\circ}\text{F}$ , and chrome-nickel steel to  $-450^{\circ}\text{F}$ .

The use of cryogenic paint removal processes on ferrous substrates, such as ship plate, may not be feasible because (in the worst case) the surface of the plate might reach  $-320^{\circ}\text{F}$ , and most shipbuilding steel is not of a type that is especially resistant to low embrittlement by very low temperature.

**Composite Materials.** The effects of very low temperature on composite materials such as glass, graphite, or boron-reinforced resin is not well known at this time. Tests of the cryogenic paint stripping process on glass-reinforced plastic and graphite composite over aluminum honeycomb core indicated no visible damage. One concern voiced by Air Force researchers is the possibility of water (condensed from the air onto the cold aircraft surface) absorbing into the matrix of a composite material. Because composite materials are not good conductors of electricity, a lightning strike on an aircraft made of composite material is not readily dissipated. If the aircraft skin contains a significant amount of water, that water could be vaporized, causing significant damage to the aircraft.

## SUMMARY AND CONCLUSIONS

The results of hundreds of detailed experiments indicate that it is not possible to remove paint from aircraft hulls using a jet of cryogenic fluid (e.g., liquid nitrogen at  $-320^{\circ}\text{F}$ ). Modern aircraft paint systems are not applied thick enough to permit establishment of a large temperature gradient across the paint film. A large temperature gradient across the paint film is one of the requirements for paint removal by thermal shock. Also, aircraft paint formulations have a coefficient of thermal expansion so close to that of aluminum that high differential stresses are not developed at the paint-substrate interface as the aircraft panel is cooled to a very low temperature.

It was demonstrated that a jet of cryogenic fluid can cause thermal shock cracking and partial removal of thick (greater than 20 mils in thickness) paint films.

It was also demonstrated that a jet of liquid nitrogen can effectively remove certain protective coatings (regardless of thickness) by differential thermal contraction. Common red oxide marine primer is one coating that is easily removed.

Although a jet of liquid nitrogen has been shown capable of removing thick paint films and marine primer coatings, the method is not recommended for application to ship hulls because of the potential for embrittlement of steel by the very low temperatures experienced in the process.

Cryogenic paint removal may have application in specific circumstances where use of conventional paint stripping methods would increase the risk of environmental damage. For example, one of the advantages of cryogenic paint stripping is that the only hazardous waste product generated by the process is paint chips, which can be easily collected using a vacuum system. Therefore, cryogenic paint stripping may be used for removal of thick layers of lead-base paint, where proper collection and management of the paint waste is essential. Use of abrasive blasting for paint removal in this situation would reduce the paint film to very small particles and disperse the particles to the atmosphere and blasting medium.

It is interesting to speculate about why cryogenic paint stripping worked on the Statue of Liberty but failed to be an effective paint removal technique in most other circumstances. References 1 and 3 indicate that the paint system on the interior of the Statue of Liberty consisted of a thick layer of old paint applied over a layer of bituminous compound. The bituminous compound was applied to the copper skin of the statue as a corrosion inhibitor. The thickness of the paint, combined with the fact that it was embrittled by age and bonded to the bituminous undercoat, resulted in a paint topcoat that was easily removed by the action of a high-pressure jet of liquid nitrogen. This paint system was modeled in the laboratory. Sample panels were coated with a thick layer of brittle epoxy paint over a layer of red oxide primer (an amorphous coating having mechanical properties similar to a bituminous coating). These panels were also easily stripped by a jet of liquid nitrogen. It should be noted that Reference 3 states that some of the bituminous undercoat on the interior of the Statue of Liberty was not removed by the liquid nitrogen jet. The jet removed only the topcoat. The bituminous undercoat was removed using an abrasive blasting system that used pellets of sodium bicarbonate as the blasting medium.

## ACKNOWLEDGMENTS

The authors wish to acknowledge the contributions made to this effort by Mr. Raymond Cappillino, Mr. Gerald Duffy, Ms. Cynthia Ruf, Mr. Daniel Zarate, and Mr. Paul Schatzberg. Mr. Cappillino assembled much of the test apparatus and assisted in conducting experiments. Mr. Duffy prepared the instrumentation for the test samples and assisted in setup of the data acquisition system. Ms. Ruf and Mr. Stone performed the

Phase I experimental work and Mr. Zarate assisted with the setup and operation of the Instron machine. The authors also wish to thank Mr. Paul Schatzberg, Navy 6.2 Block Program Manager for Environmental Protection, for his guidance and support.

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Table 1. Properties of Paints Used in Experiments

| Paint or Prepared Sample   | Source   | Thickness (mils) | $\alpha$ (in./in./°F) |
|--|--|------------------|-----------------------|
| Primer chromate, water reducible epoxy aircraft primer, MIL-P-85582A, type II, Class 1                     | Pt. Mugu Missile Test Center, CA, paint shop                               | 1                | Not measured          |
| Topcoat epoxy-polyamide, MIL-C-22750D, gray 16440, type 1  | Pt. Mugu, CA   | 4 to 20          | 9.5 E-06              |
| Topcoat epoxy-polyamide color 36320, type 1  | Pt. Mugu, CA   | 3 to 4           | 11.8 E-06             |
| Surface prep: MIL-C-5541, Primer: polyurethane MIL-P-85853 Topcoat: polyurethane MIL-C-83286               | Sample from NADEP, Jacksonville, FL  | 2 to 4           | 6.3 E-06              |
| Surface prep: MIL-C-5541, Primer: epoxy, type 1 MIL-P-85582, Topcoat: high solids polyurethane MIL-C-25582 | Sample from NADEP, JAX   | 4 to 5           | 12 E-06               |
| Unknown  | Sample of graphite fiber composite over aluminum honeycomb from NADEP, JAX | 6                | 5.6 E-06              |
| "Unicoat"  | Sample of new "one coat" paint from NADC, Warminster, PA                   | 2                | Not measured          |

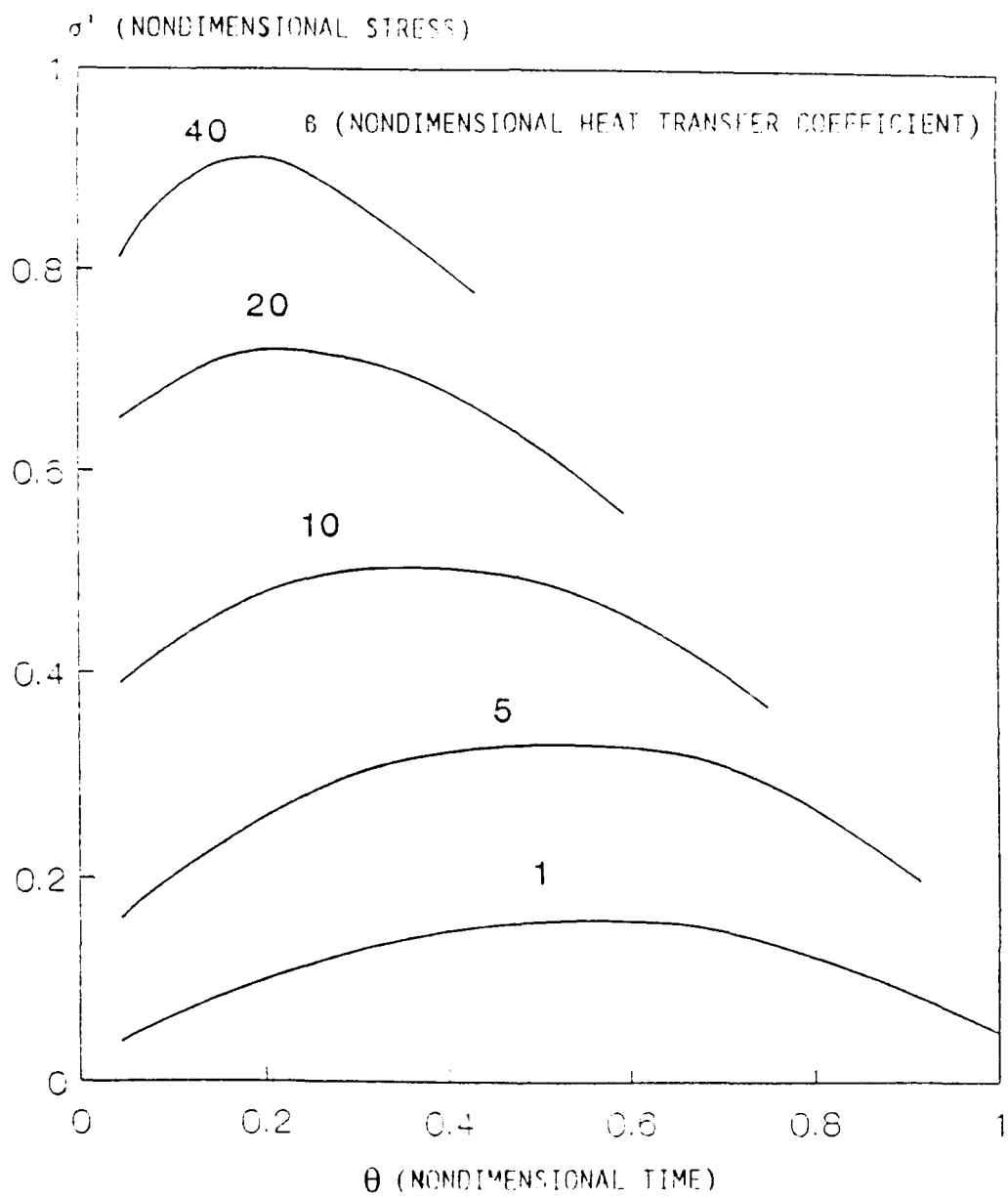


Figure 1. Surface stress solution flat plates

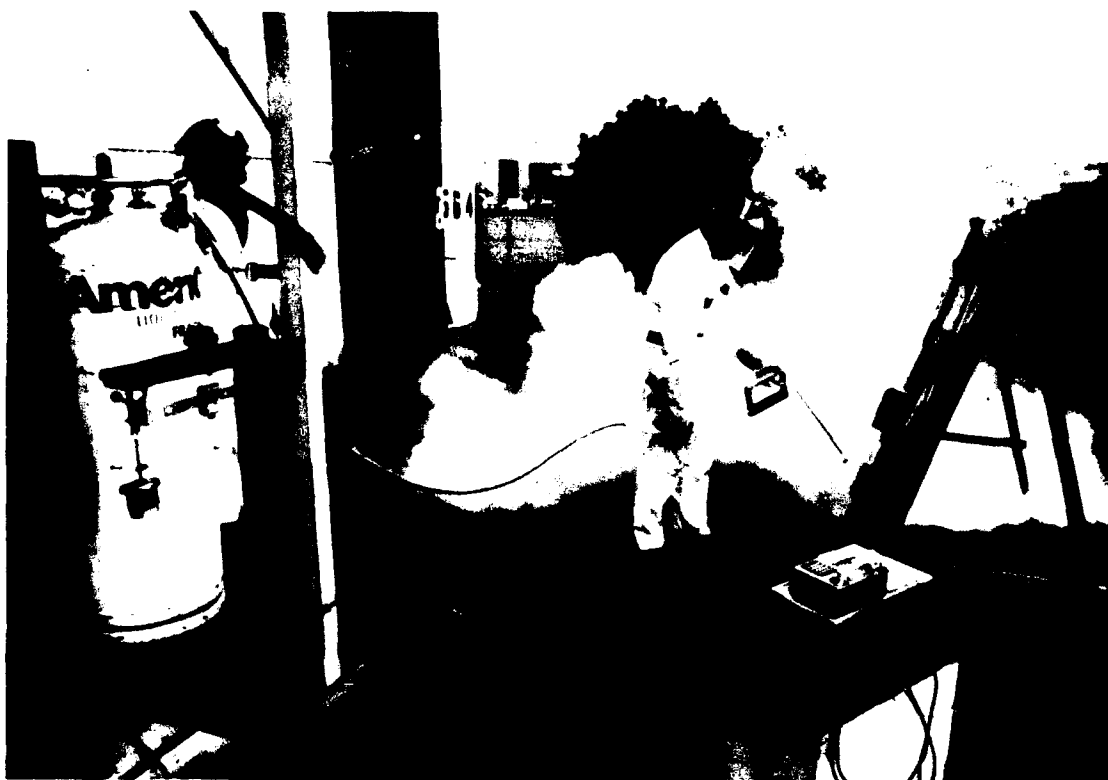


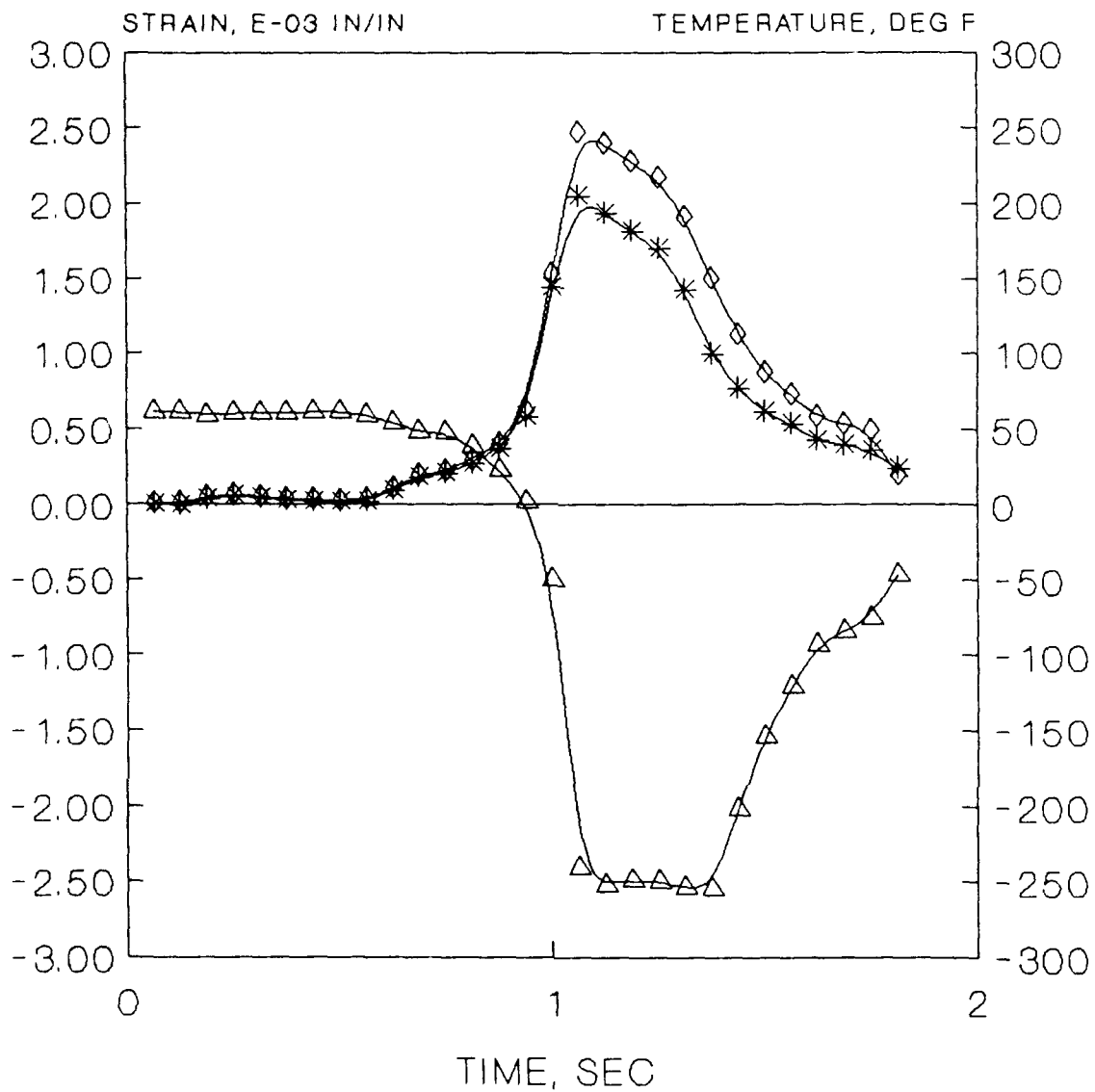
Figure 2. Low-pressure, hand-held liquid nitrogen applicator.



Figure 3. Paint damage caused by application of liquid nitrogen.



Figure 4. Automated liquid nitrogen applicator system.

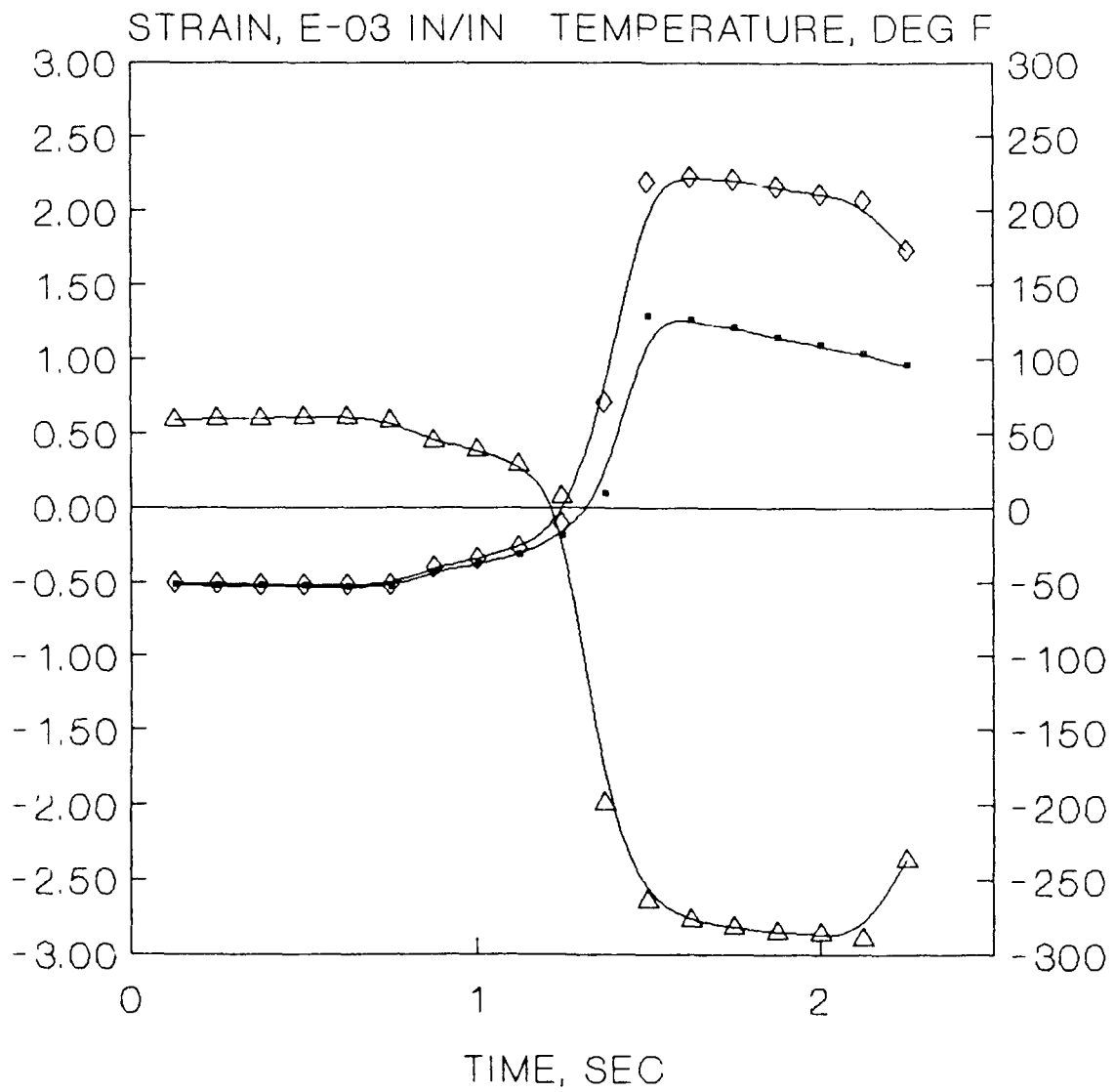


\*— INDICATED STRAIN    ♦— CORRECTED STRAIN  
 —△— TEMPERATURE

PAINT SURF, ALUMINUM PLATE  
 SPEED = 6 in/sec, P = 165 psi, SO = 1 in

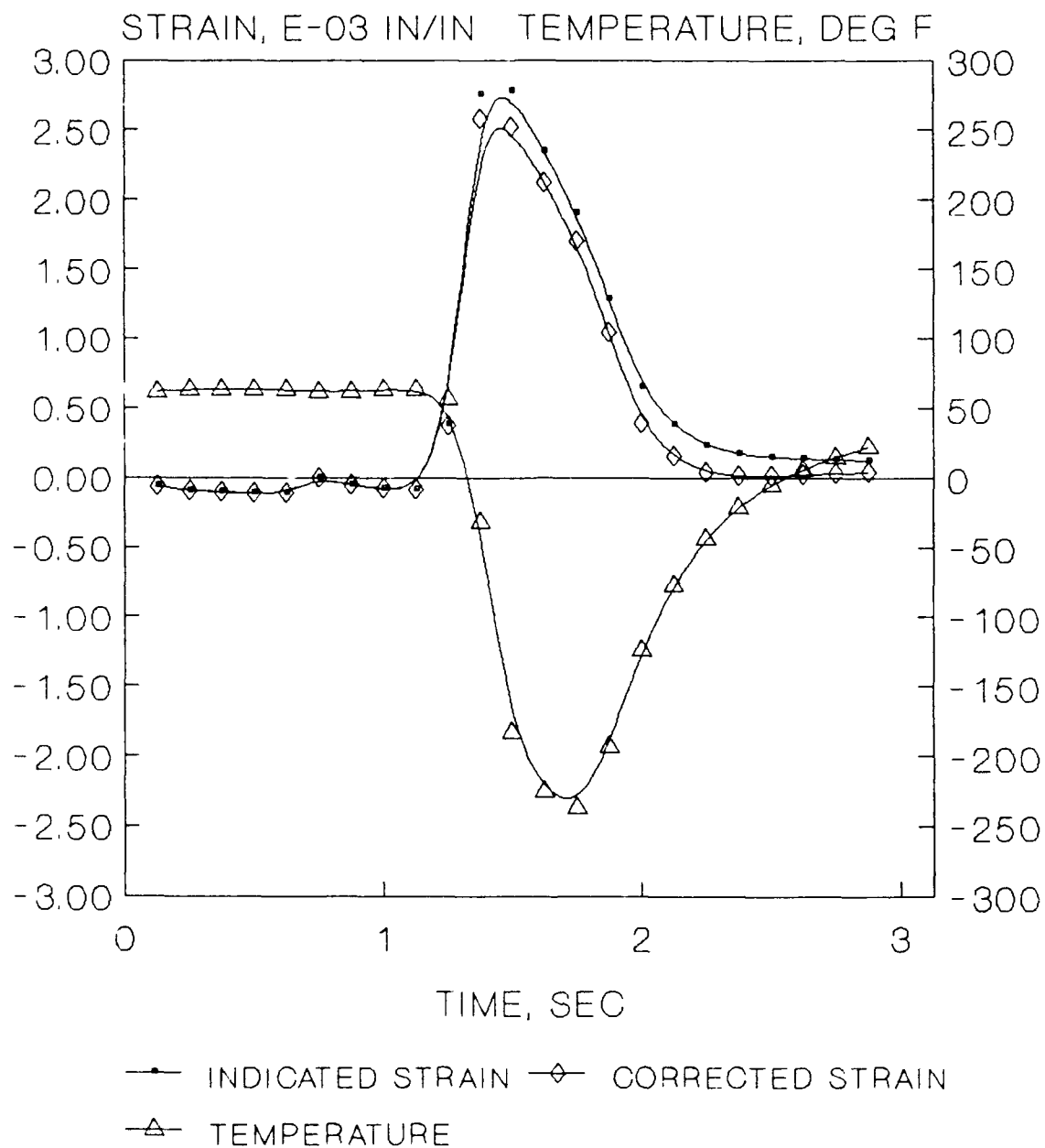
Figure 5. Strain and temperature history from Test Q25.





PAINT SURF, FRP COMPOSITE  
SPEED = 6, ANGLE = 30, P = 160

Figure 6. Strain and temperature history from Test E13.



PAINT SURF, STEEL PLATE  
SPEED = 6, P = 186 psl, ANGLE = 60

Figure 7. Strain and temperature history from Test J15.

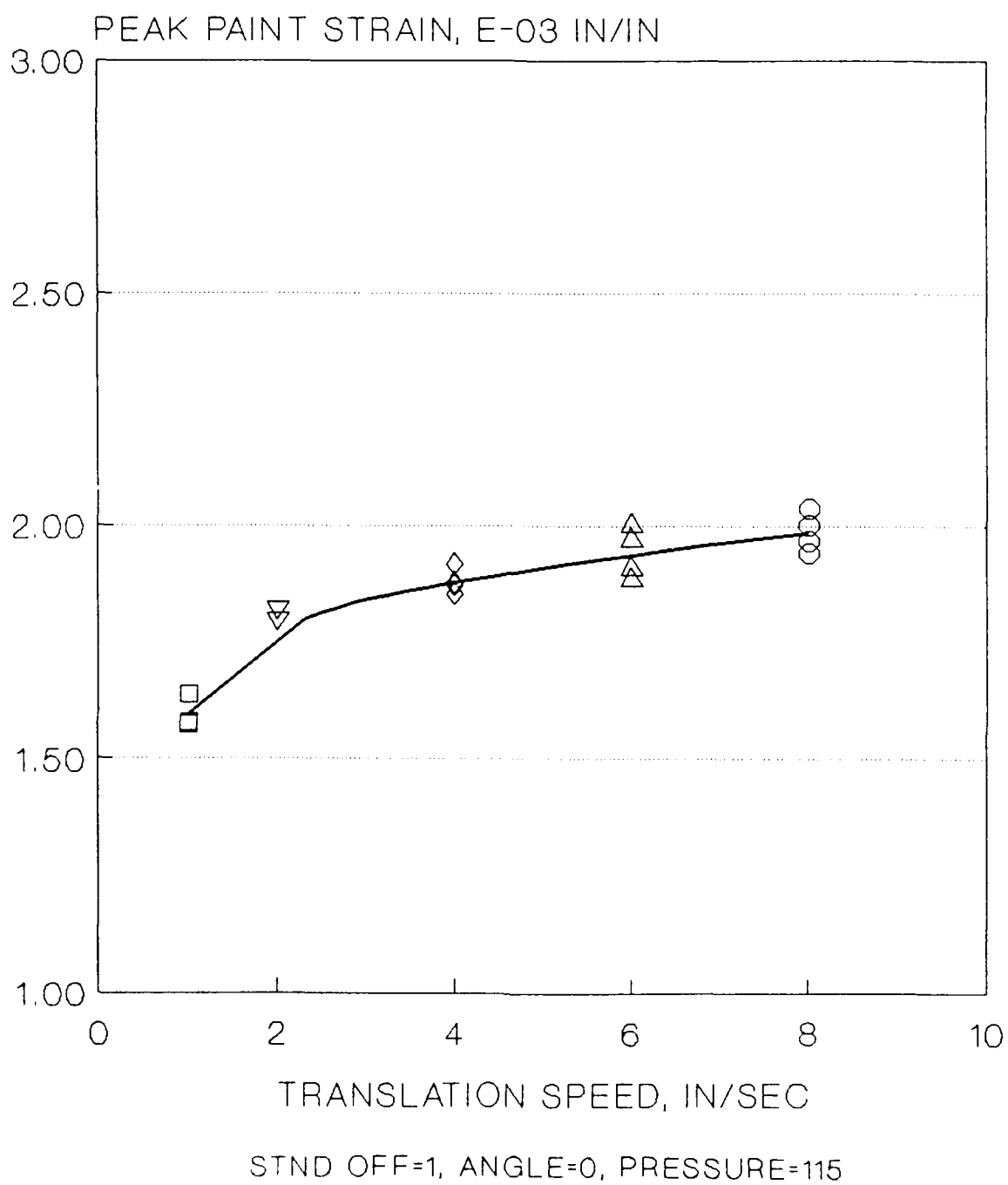


Figure 8. Peak strain versus speed.

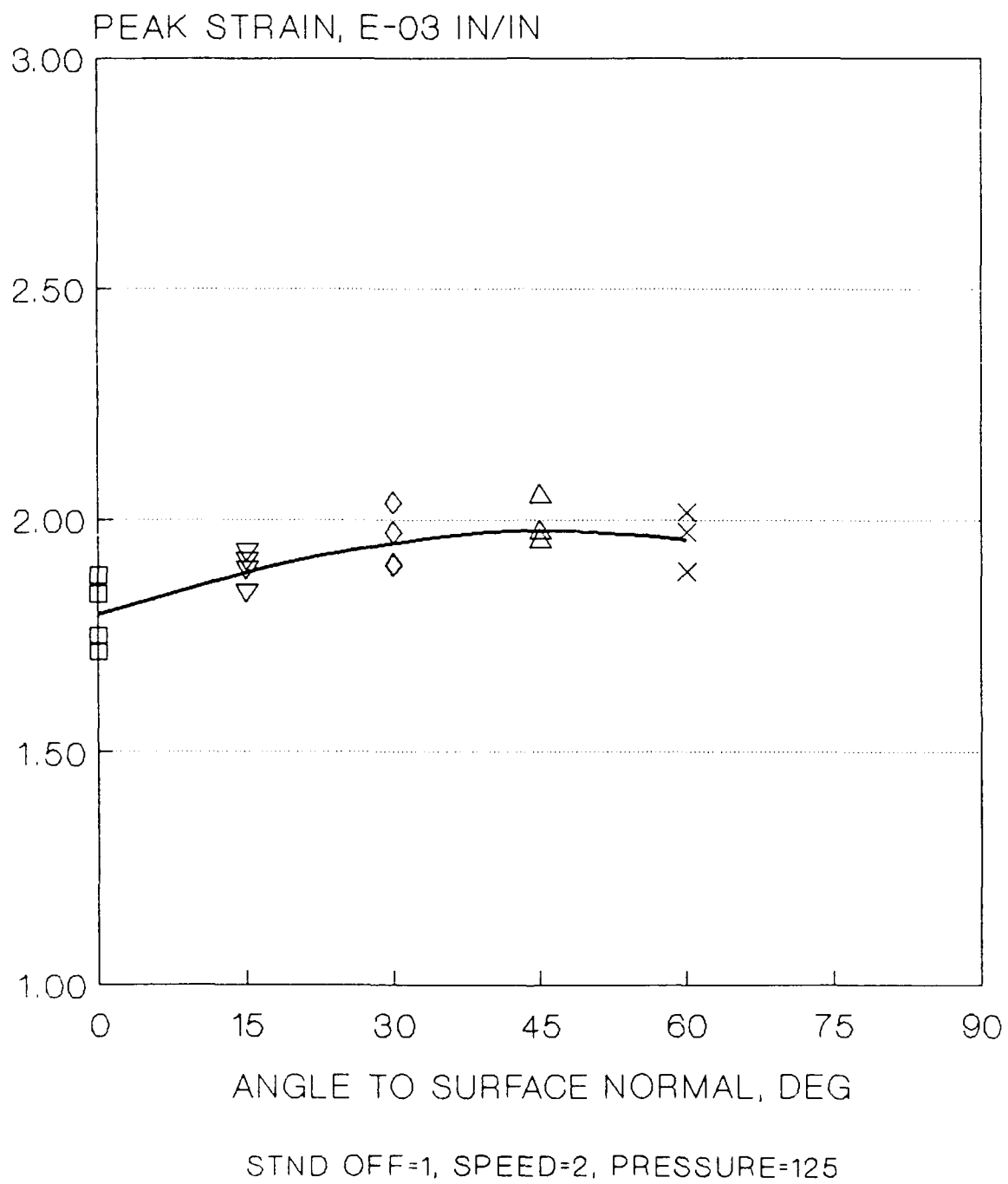
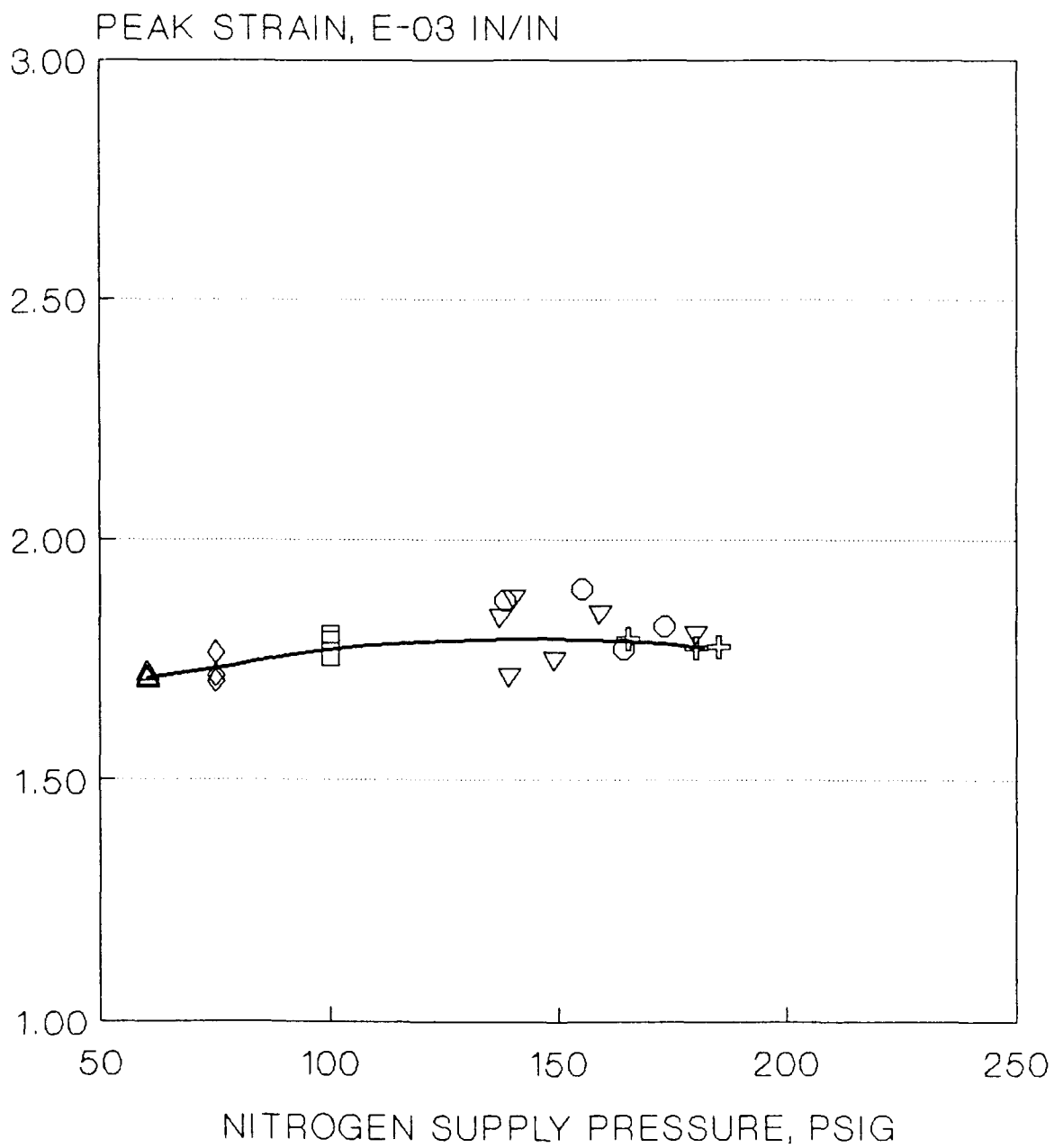
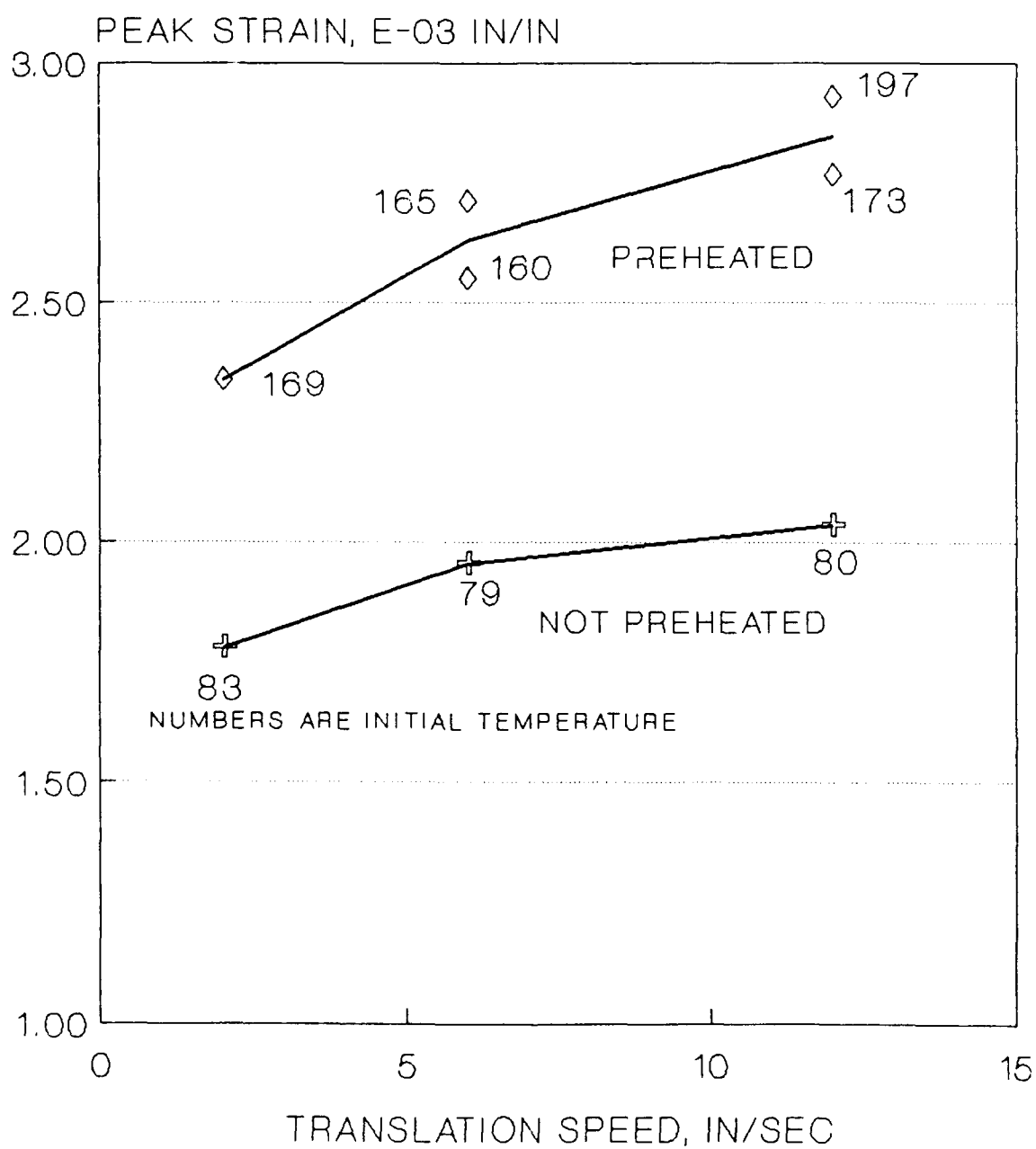


Figure 9. Peak strain versus angle



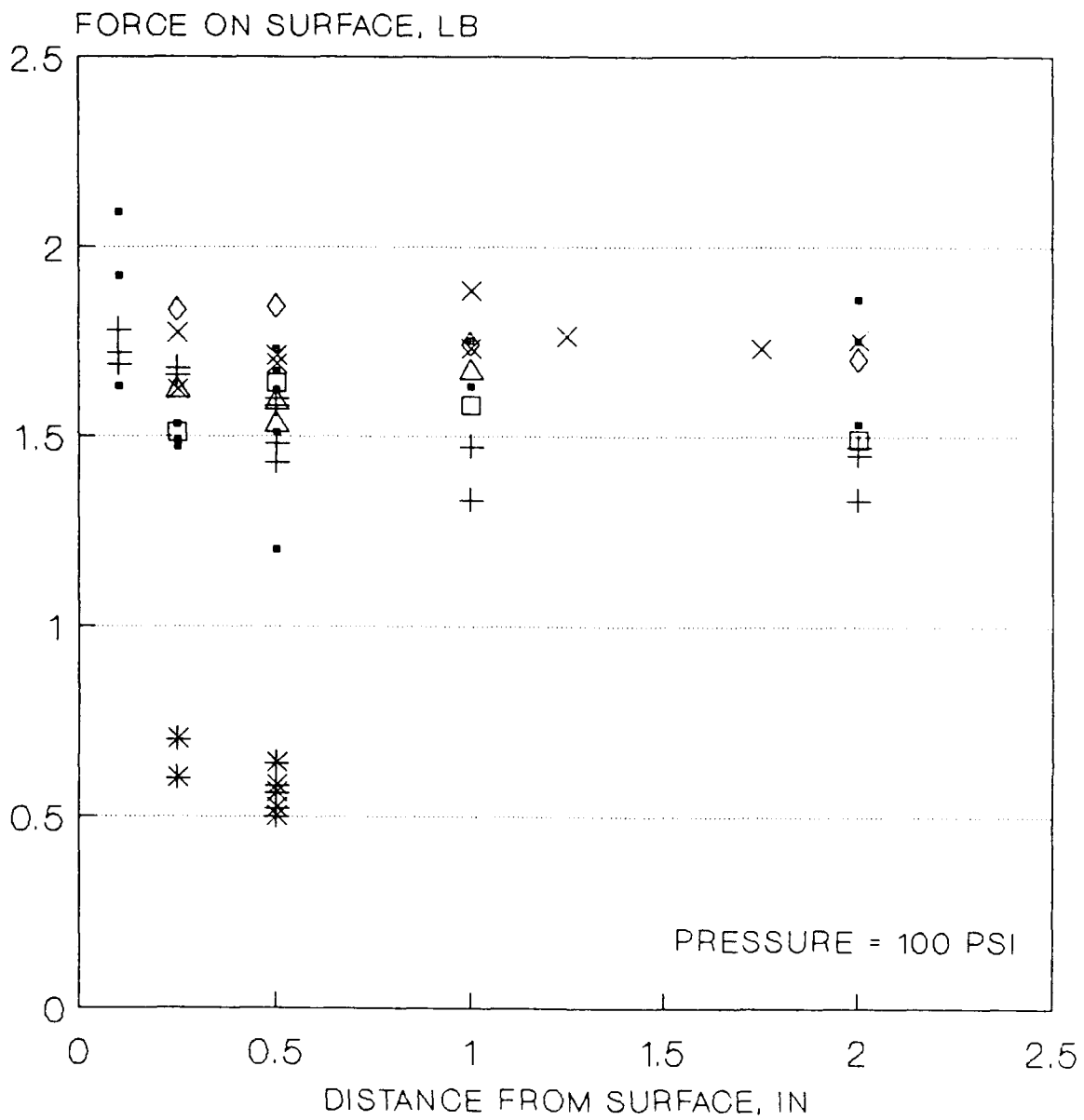
STND OFF=1, SPEED=2, ANGLE=0

Figure 10. Peak strain versus pressure.



STND OFF=1, ANGLE=30, PRESS=125, DIA=.18

Figure 11. Sensitivity to initial temperature.



DIFFERENT SYMBOLS DESIGNATE DIFFERENT NOZZLE DESIGNS

Figure 12. Normalized jet force.

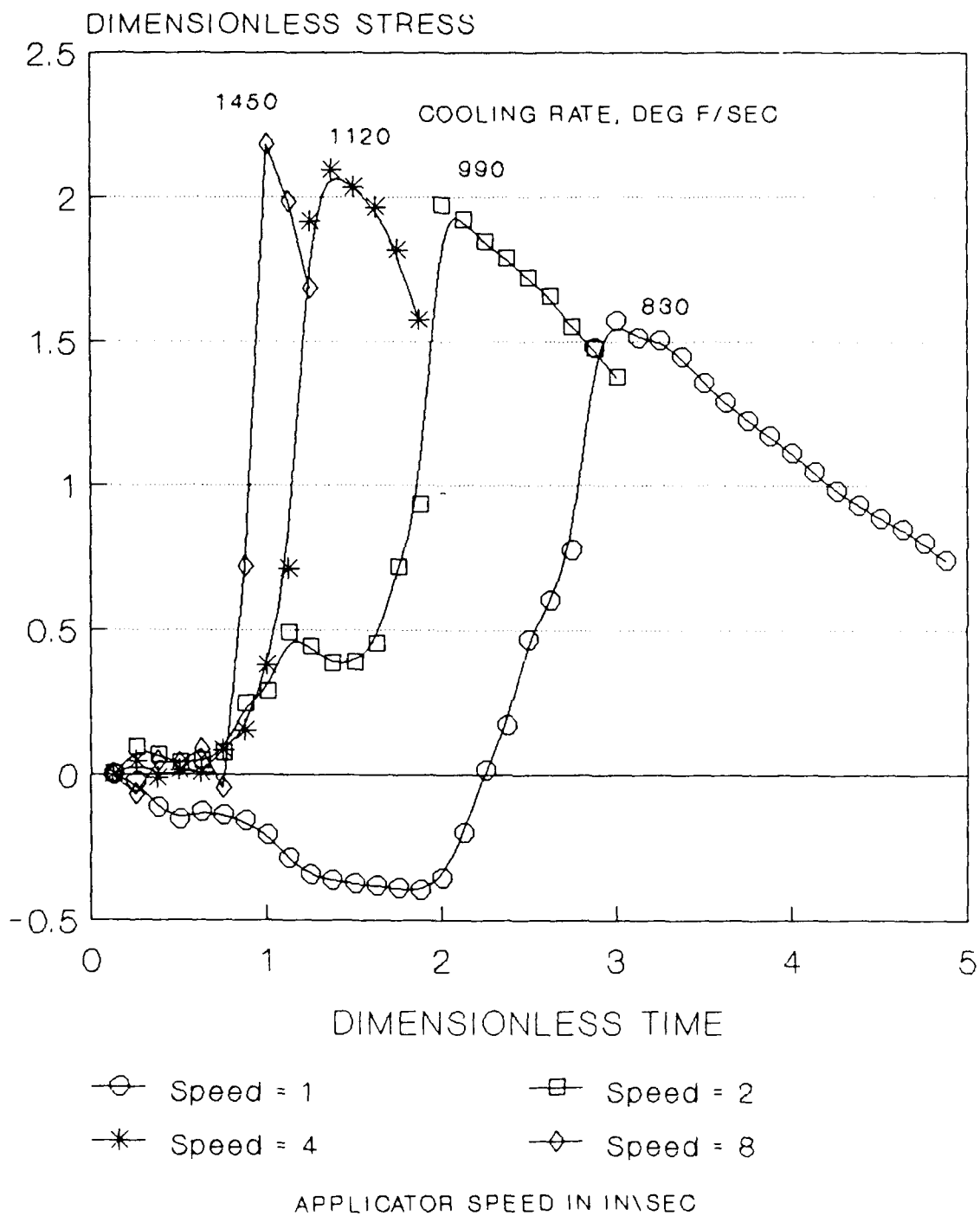


Figure 13. Dimensionless stress and time.



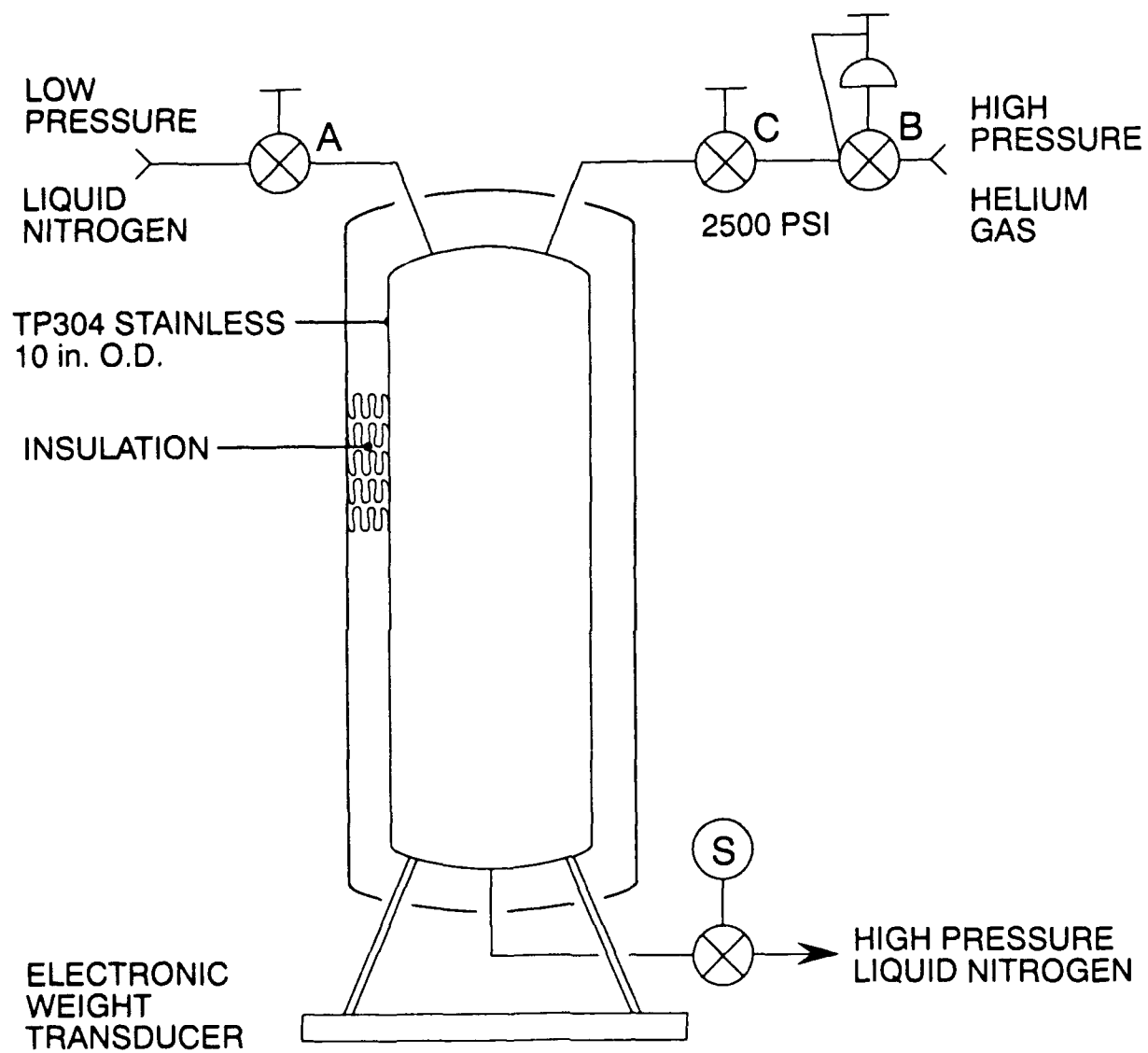


Figure 14. Schematic of high-pressure liquid nitrogen applicator.

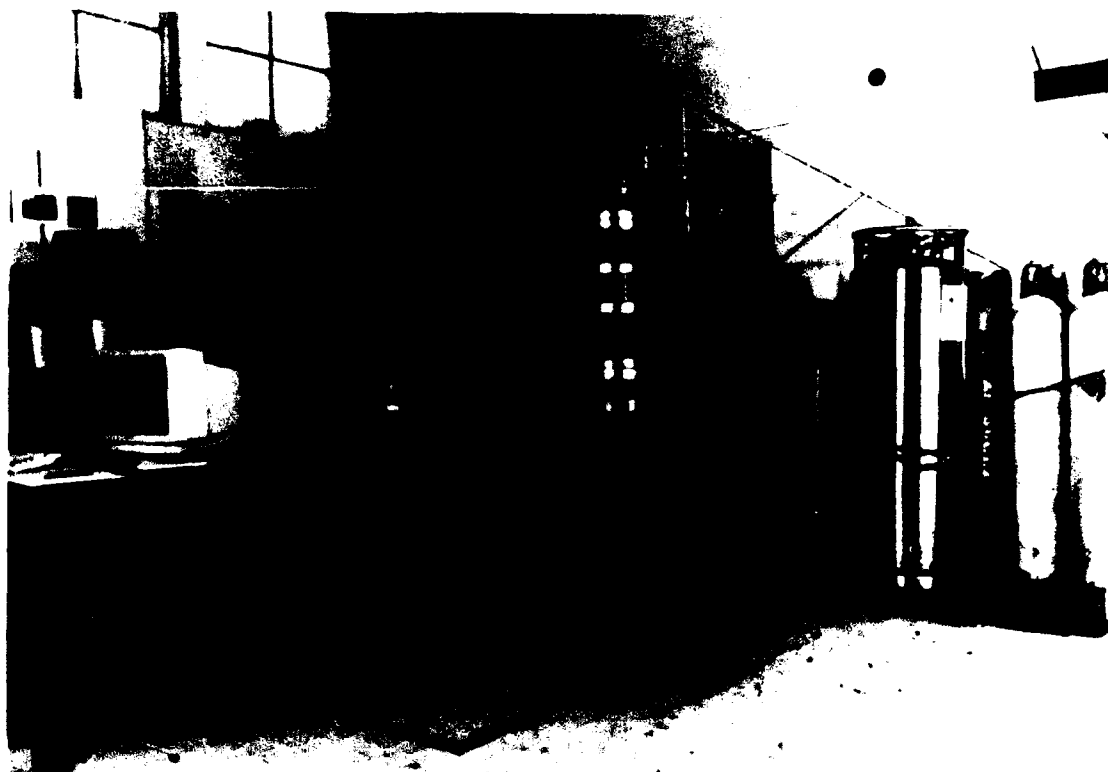
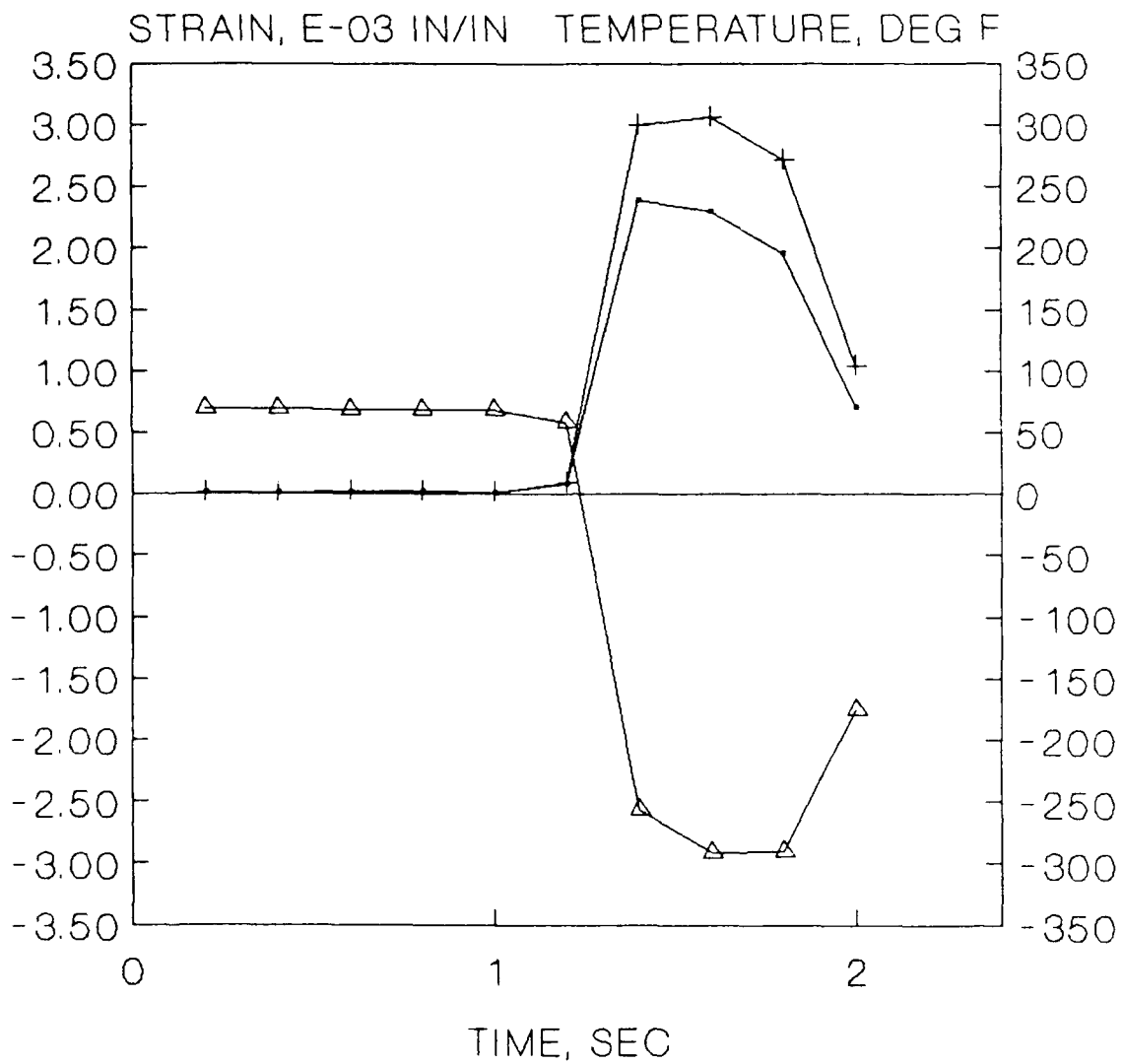


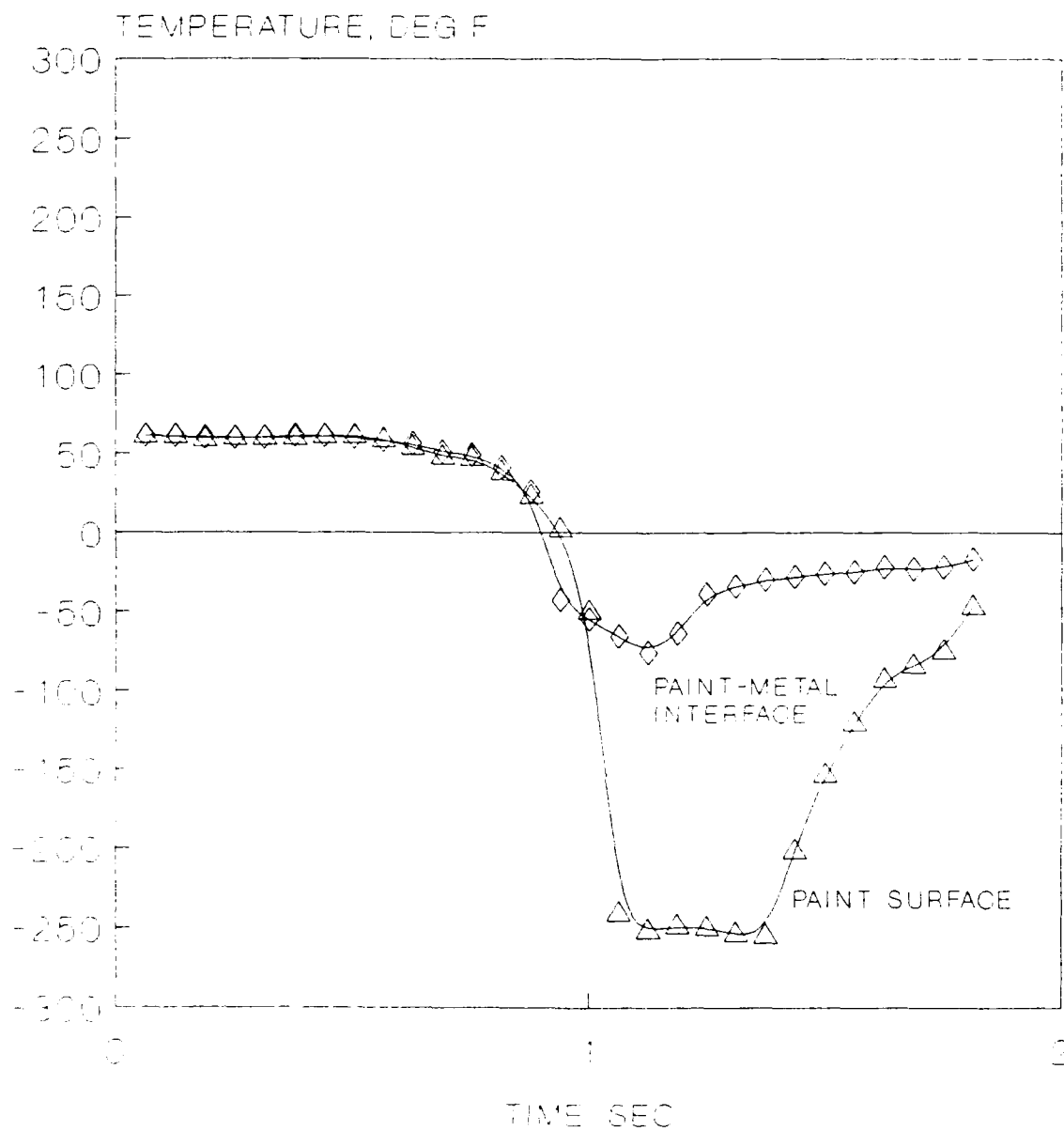
Figure 15. Photograph of high-pressure liquid nitrogen applicator.



—•— INDICATED STRAIN —+— CORRECTED STRAIN  
 —△— TEMPERATURE

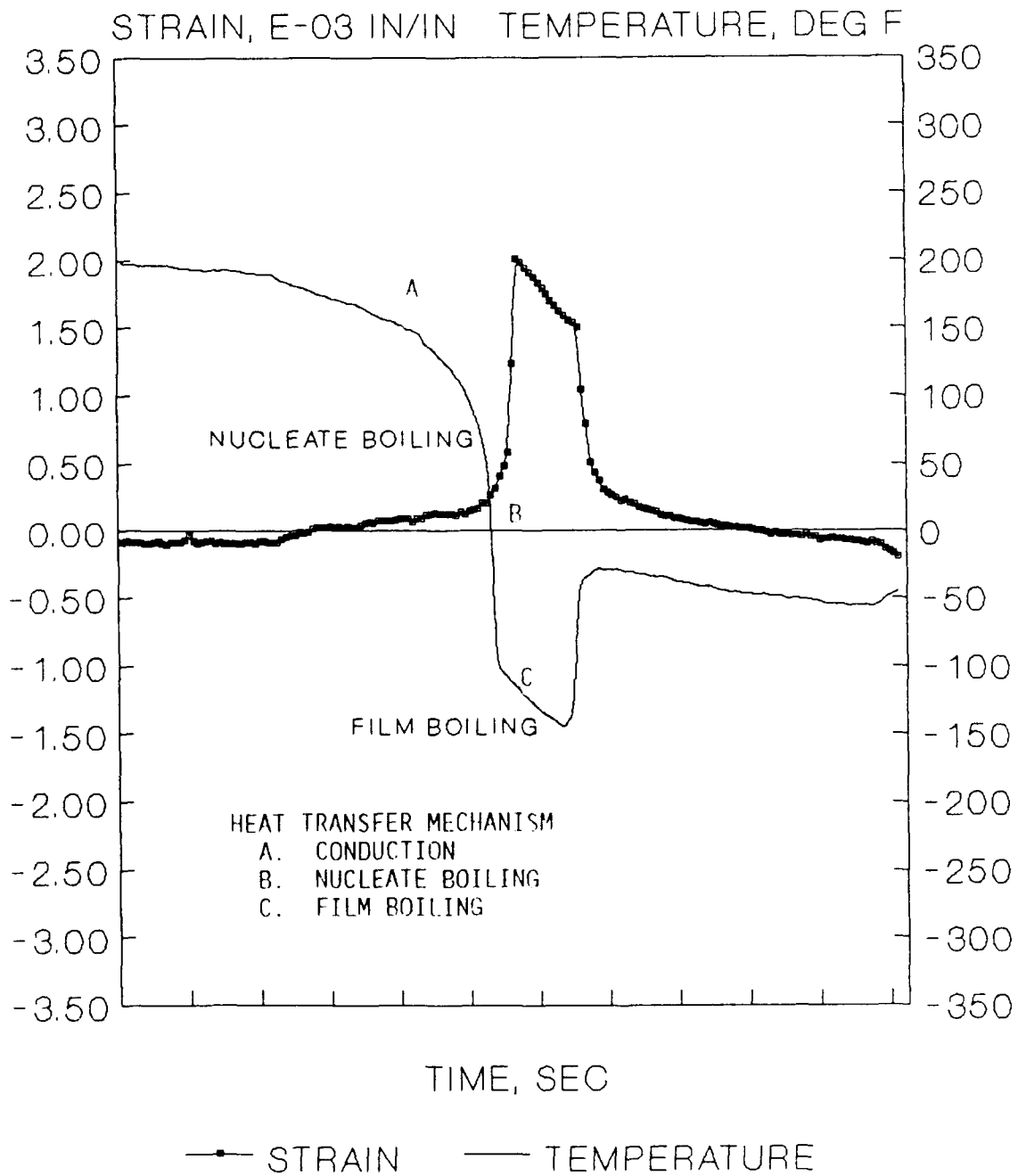
PAINT SURF, STEEL PLATE  
 P = 2250 PSI, ANGLE = 65  
 SPEED = 8 IN/SEC

Figure 16. Strain and temperature history from Test HP C2.



ALUMINUM PLATE SPEED = 6 IN/SEC  
P = 165 PSI, SD = 1 IN

Figure 17. Temperature comparison.



$T_0 = 200$  DEG F, SPD = 1, P = 230

Figure 18. Strain versus temperature.

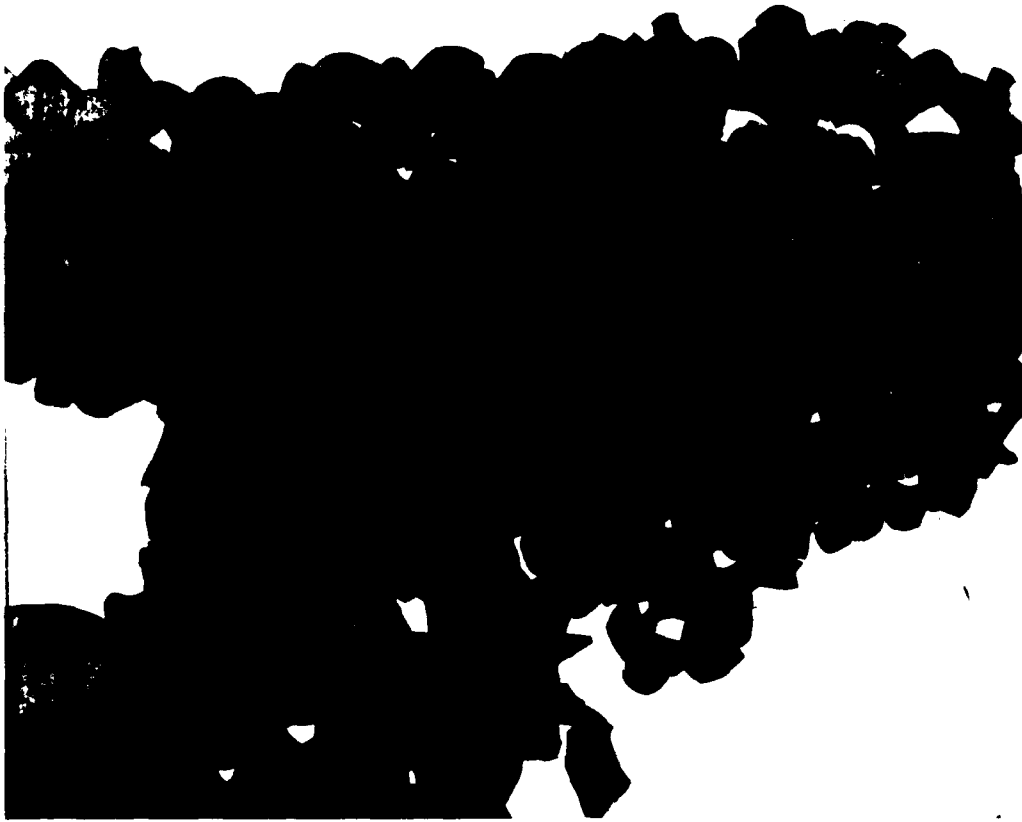


Figure 19. Photo of paint removed by the mechanism of thermal contraction.

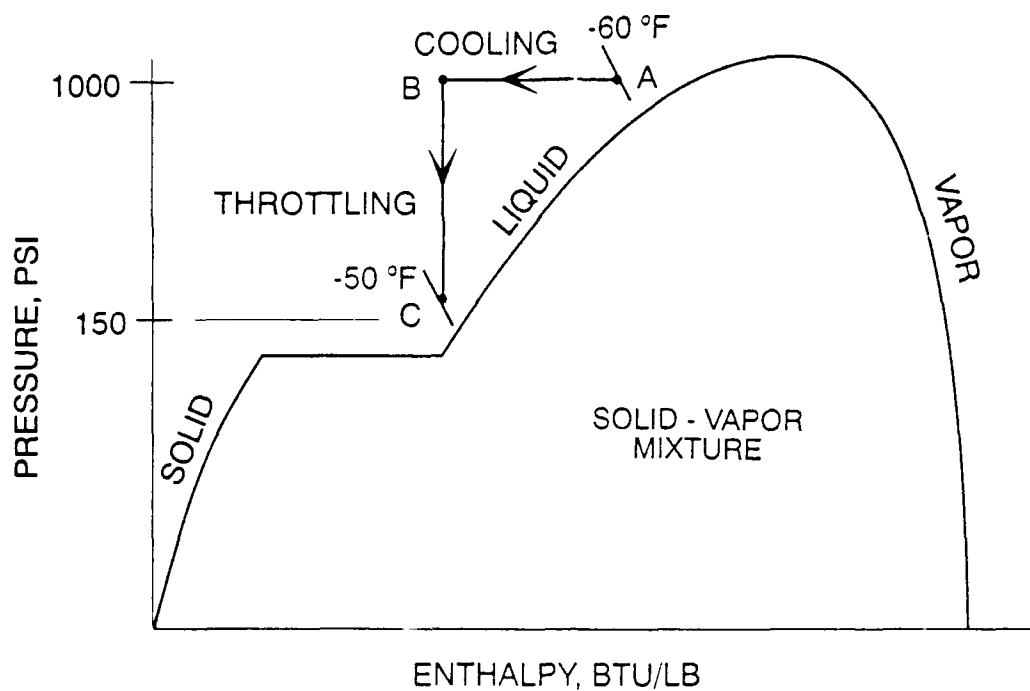


Figure 20. Pressure-enthalpy diagram for CO<sub>2</sub>.

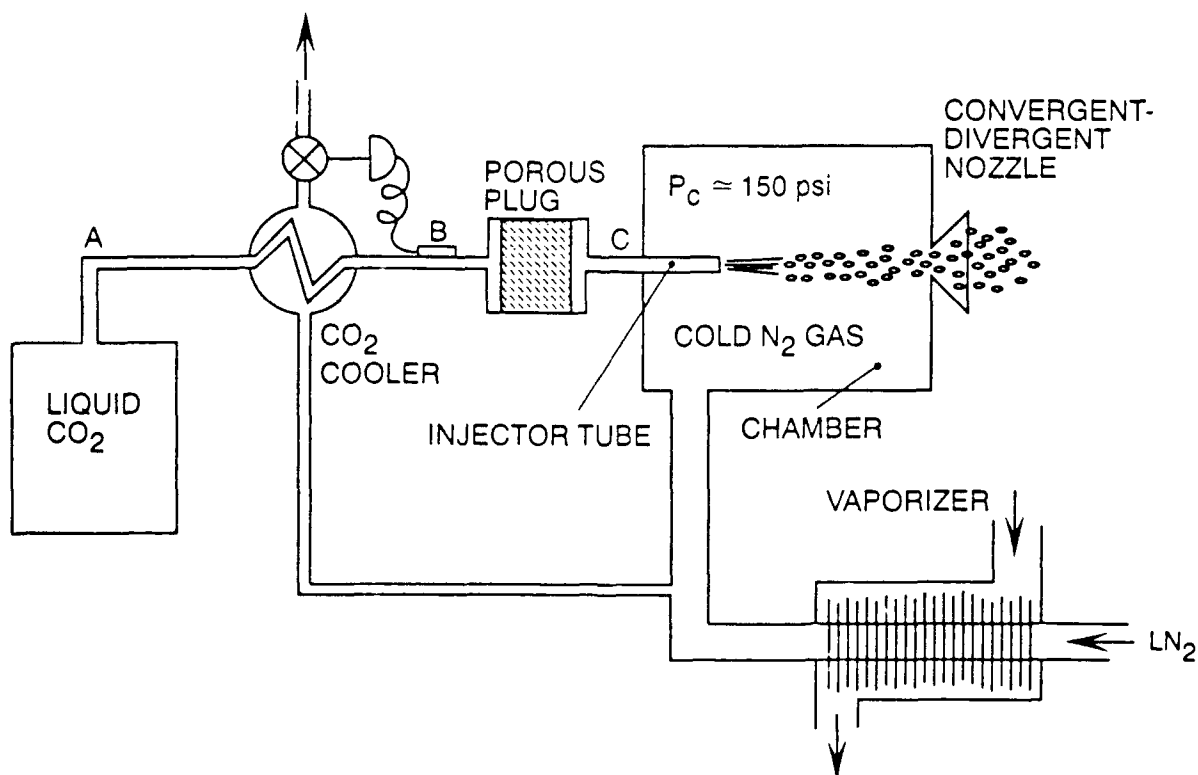


Figure 21. Schematic of CO<sub>2</sub> particle generator

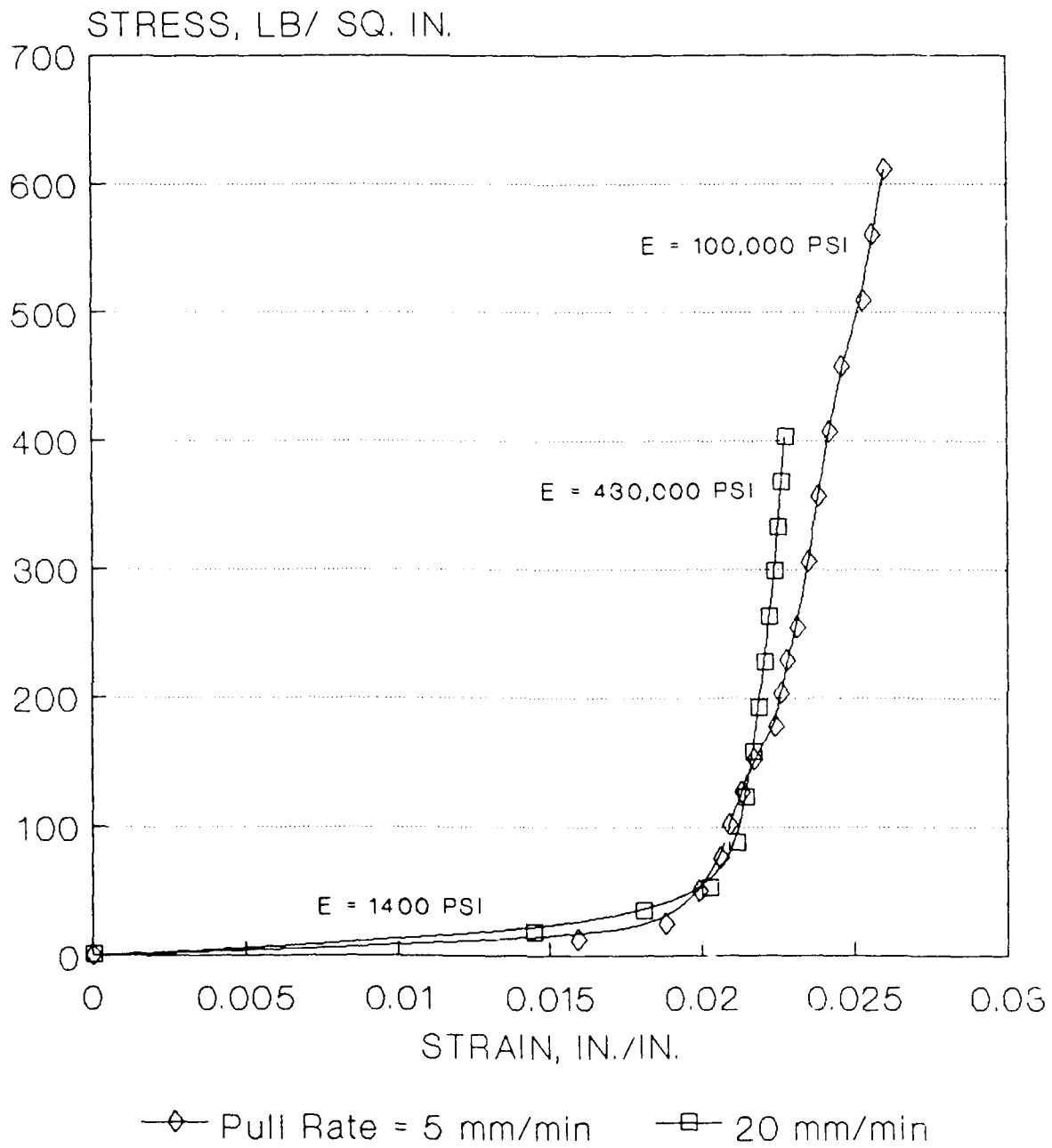


Figure 22. Stress-strain data for paint.



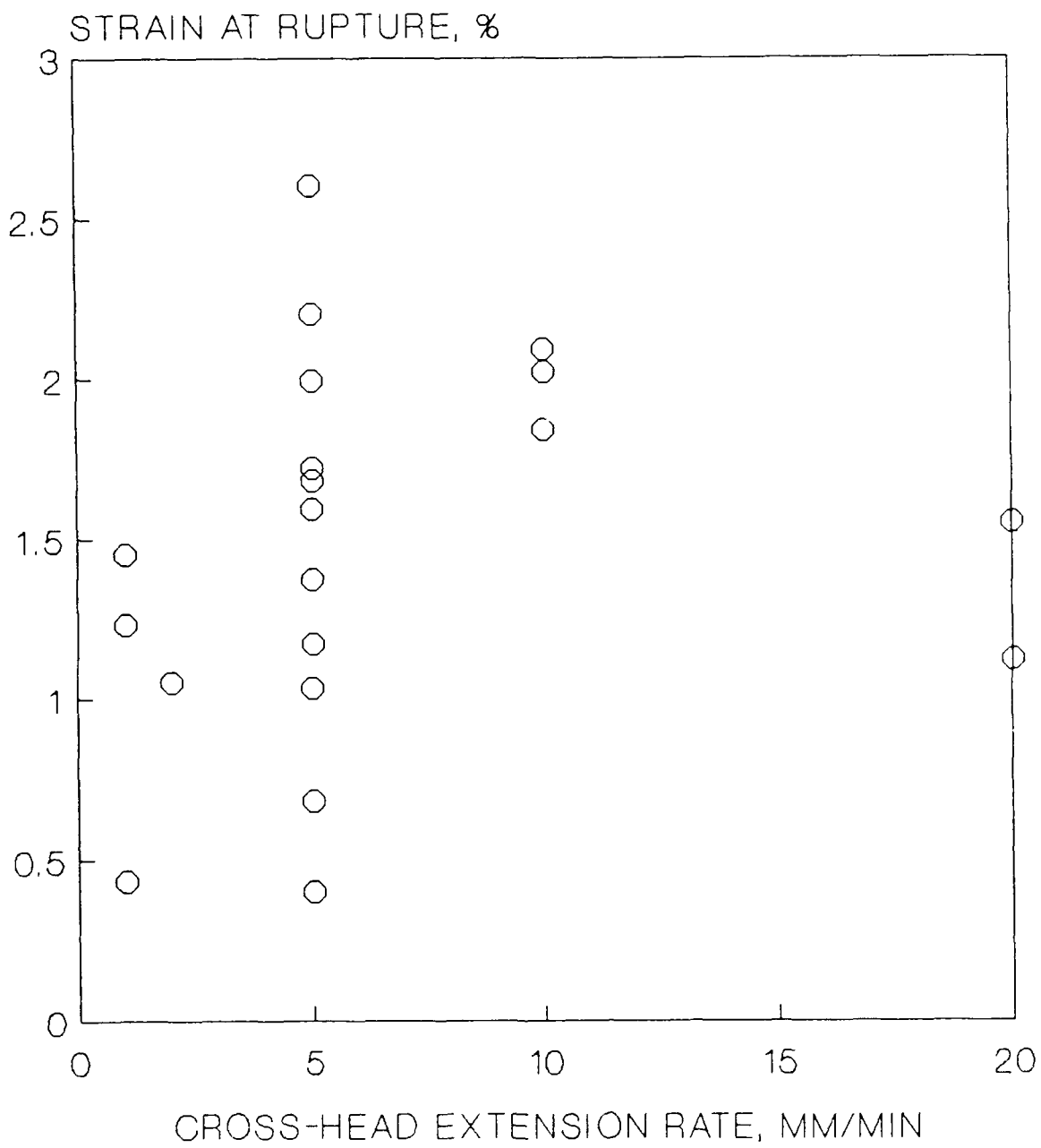


Figure 23. Tensile test results for paint.

## Appendix

### CORRECTIONS FOR TEMPERATURE-INDUCED APPARENT STRAIN AND GAGE FACTOR VARIATIONS IN STRAIN GAGES

Ideally, an electrical resistance strain gage bonded to a test panel would respond only to the applied strain on the part and would not be affected by other variables. Unfortunately, the electrical resistance of a strain gage changes not only with strain, but also with temperature. In addition, the relationship between strain and electrical resistance, called the gage factor, is also a function of temperature. Finally, the difference between the coefficients of thermal expansion of the gage material and the surface to which it is bonded also produces a temperature-induced apparent strain. This temperature-dependent behavior can cause significant errors in data interpretation in this experimental work because the temperature change is large and the magnitude of the measured strain is small. Fortunately, these errors can be controlled or eliminated by application of compensation factors.

The true strain at the test temperature, corrected for thermal effects, is obtained as follows:

$$\begin{aligned}\epsilon_{\text{true}} = & [\epsilon_{\text{indic}} - \epsilon_{\text{appar}}(T)] \cdot F'/F(T) \\ & - (\alpha_s - \alpha_g) \cdot (T_{\text{ref}} - T)\end{aligned}\quad (\text{A-1})$$

where:  $\epsilon_{\text{true}}$  = the corrected strain, in./in.

$\epsilon_{\text{indic}}$  = indicated strain at test temperature T

= strain value recorded by data logger, in./in.

$\epsilon_{\text{appar}}(T)$  = apparent strain at test temperature

= apparent strain due to change in resistivity  
with temperature (supplied by gage manufacturer)

=  $-89.6 + 1.53 \cdot T - 4.75 \text{ E-}03 \cdot T^2$   
 $+ 4.33 \text{ E-}06 \cdot T^3$ ,  $\mu\text{in./in.}$

$F'$  = gage factor at room temperature (supplied by  
manufacturer)

$F(T)$  = gage factor at test temperature (supplied by  
manufacturer)

=  $-9.44 \text{ E-}03 \cdot (T - T_{\text{ref}})$

$\alpha_s$  = coefficient of thermal expansion of substrate material, in./in./°F

$\alpha_g$  = coefficient of thermal expansion of gage material

= 13 E - 06 in./in./°F

$T_{ref}$  = reference temperature, °F

$T$  = test temperature, °F

The temperature-induced apparent strain can be significant compared to the magnitude of the indicated strain: as large as 50 percent of indicated strain in some experiments.

Equation A-1 suggests a method whereby the coefficient of thermal expansion of a material can be measured. If an instrumented sample of material is under no mechanical stress and is at thermal equilibrium (i.e., its temperature is constant), then the true strain ( $\epsilon_{true}$ ) must equal zero. Therefore,

$$\alpha_s = \alpha_g + [\epsilon_{indic} - \epsilon_{appar}(T)] \cdot F' / [F(T) \cdot (T_{ref} - T)] \quad (A-2)$$

All terms on the right side of Equation A-2 are either available from the strain gage manufacturer or easily measured.

Strain gages and thermocouples were bonded to small (1-inch square) samples of painted substrate material then lowered into a Dewar of liquid nitrogen. When the temperature of the sample reached equilibrium, the indicated strain was recorded. The apparent coefficient of thermal expansion of the coating was then determined using Equation A-2. This value of  $\alpha_s$  was then input to the computer programs used to analyze and display the experimental data.

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#### 1 SHORE FACILITIES

- 1A Construction methods and materials (including corrosion control, coatings)
- 1B Waterfront structures (maintenance/deterioration control)
- 1C Utilities (including power conditioning)
- 1D Explosives safety
- 1E Aviation Engineering Test Facilities
- 1F Fire prevention and control
- 1G Antenna technology
- 1H Structural analysis and design (including numerical and computer techniques)
- 1J Protective construction (including hardened shelters, shock and vibration studies)
- 1K Soil/rock mechanics
- 1L Airfields and pavements
- 1M Physical security

#### 2 ADVANCED BASE AND AMPHIBIOUS FACILITIES

- 2A Base facilities (including shelters, power generation, water supplies)
- 2B Expedient roads/airfields/bridges
- 2C Over-the-beach operations (including breakwaters, wave forces)
- 2D POL storage, transfer, and distribution
- 2E Polar engineering

#### 3 ENERGY/POWER GENERATION

- 3A Thermal conservation (thermal engineering of buildings, HVAC systems, energy loss measurement, power generation)
- 3B Controls and electrical conservation (electrical systems, energy monitoring and control systems)
- 3C Fuel flexibility (liquid fuels, coal utilization, energy from solid waste)

- 3D Alternate energy source (geothermal power, photovoltaic power systems, solar systems, wind systems, energy storage systems)

- 3E Site data and systems integration (energy resource data, integrating energy systems)

- 3F EMCS design

#### 4 ENVIRONMENTAL PROTECTION

- 4A Solid waste management
- 4B Hazardous/toxic materials management
- 4C Waterwaste management and sanitary engineering
- 4D Oil pollution removal and recovery
- 4E Air pollution
- 4F Noise abatement

#### 5 OCEAN ENGINEERING

- 5A Seafloor soils and foundations
- 5B Seafloor construction systems and operations (including diver and manipulator tools)
- 5C Undersea structures and materials
- 5D Anchors and moorings
- 5E Undersea power systems, electromechanical cables, and connectors
- 5F Pressure vessel facilities
- 5G Physical environment (including site surveying)
- 5H Ocean-based concrete structures
- 5J Hyperbaric chambers
- 5K Undersea cable dynamics

#### ARMY FEAP

- BDG Shore Facilities
- NRG Energy
- ENV Environmental/Natural Responses
- MGT Management
- PRR Pavements/Railroads

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